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THE Journal OF THE AMERICAN LEATHER CHEMISTS ASSOCIATION

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The Journal

of the

AMERICAN LEATHER CHEMISTS ASSOCIATION

Vol. LIII JUNE 1958 No. 6

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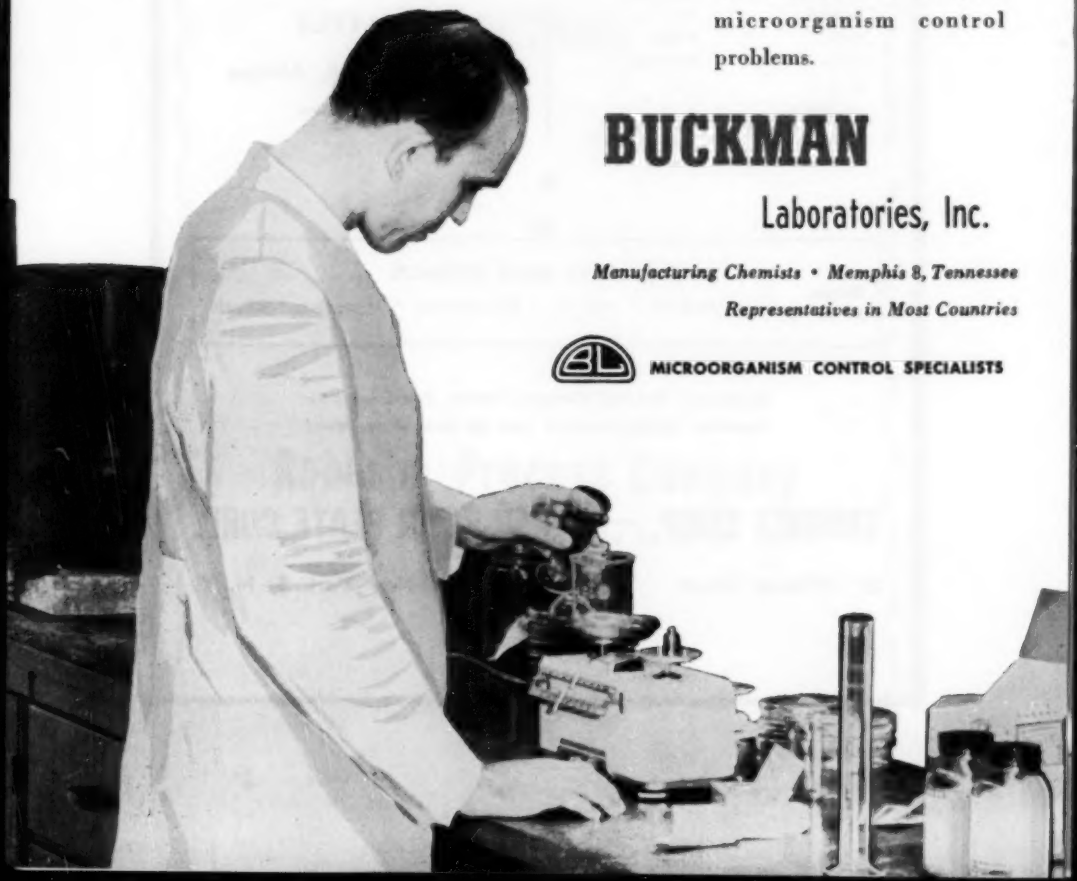

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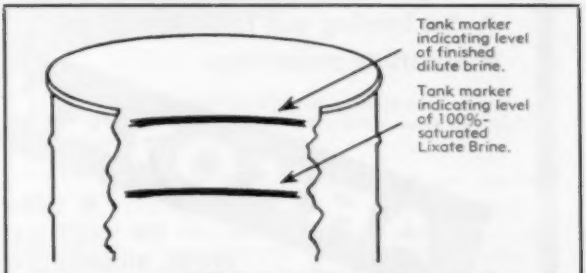

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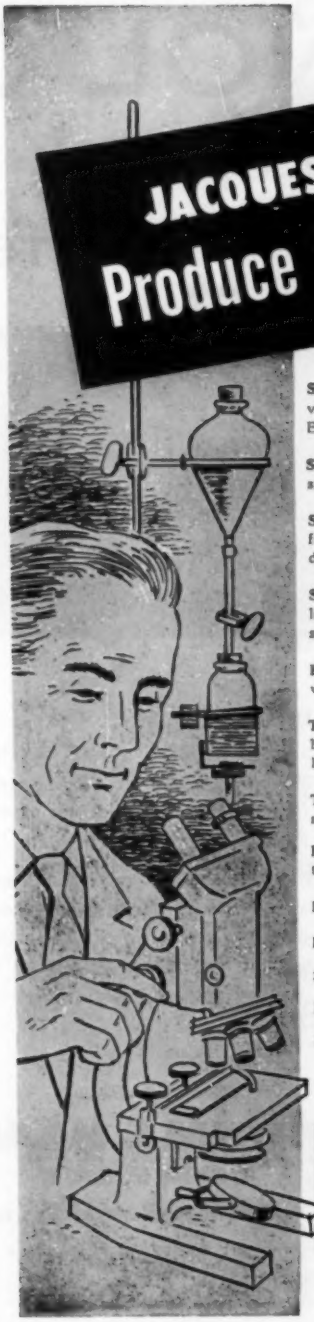

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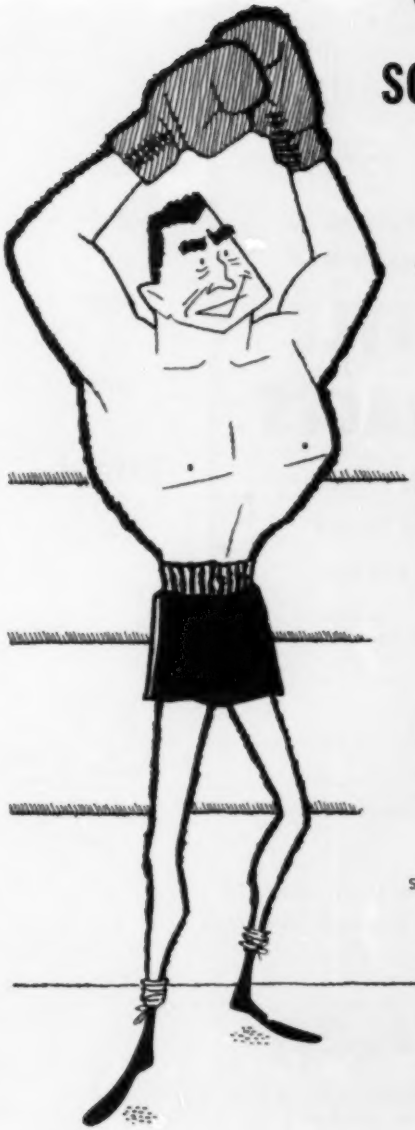

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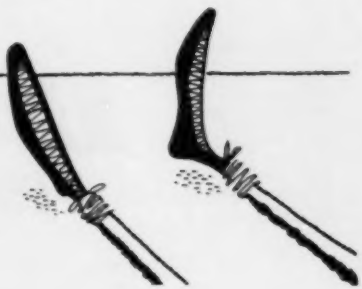

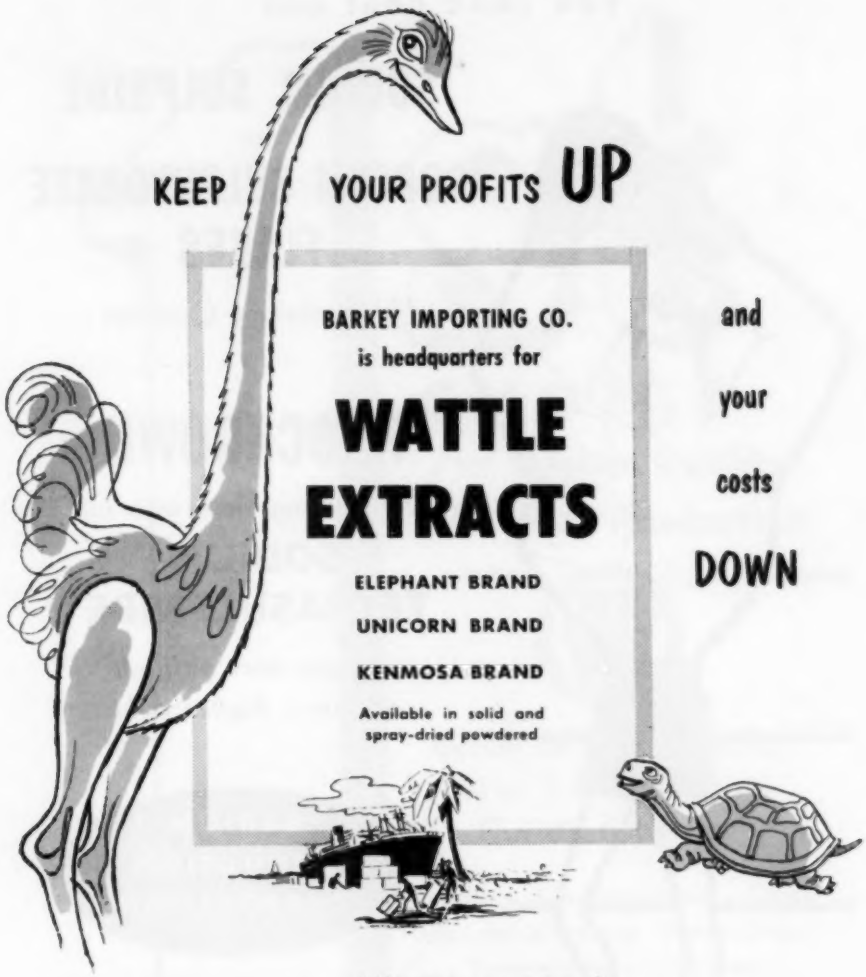

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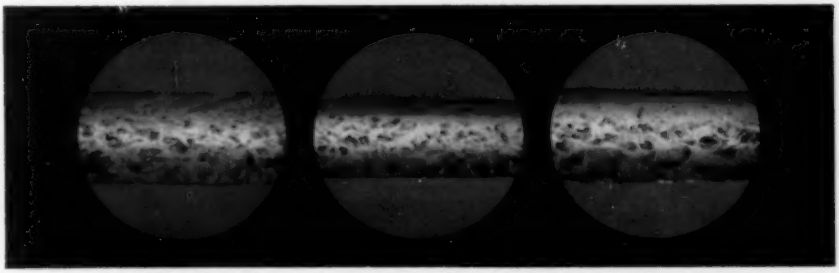

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PRODUCTION OF LEATHER USING NONAQUEOUS SYSTEMS*

ALEXIS E. USHAKOFF

Consulting Engineer
Beverly, Massachusetts

ABSTRACT

A summary of the investigations of reactions of hides in solvent systems and a description of the Secotan process for making leather in a solvent medium are given. Solvent dehydration, the predominant factors controlling leather quality, and the reactions of hides in solvents and water are discussed. Solvent dehydration involves removing water from the hydrated structure without causing shrinkage. Quality depends on the stresses and tensioning in the fiber structure produced by the degree of hydration and by high-volume materials combined with the hide substance. The reactions of hides are intimately associated with the nature of the medium in which the reaction takes place. The magnitude and direction of a reaction can be reversed or changed by a change in the medium or its composition. It is concluded that the reactions are adsorption effects. The Secotan process consists of an aqueous pretreatment followed by a solvent process in an automatic machine. The Secotan machine consists of an endless conveyor running through a series of enclosed chambers. Pretreated hides are slicked out on the loading end of the conveyor. Each hide is successively dehydrated, tanned, and/or fatliquored and dyed, and dried. It emerges from the machine flat, dry, and ready for sorting and finishing. The machine processes up to sixty sides per hour. The solvent (acetone) is automatically recovered and reused.

—⁂—

INTRODUCTION

Early in the latter part of the nineteenth century interest was developing in the possibility of employing liquids other than water as vehicles for the introduction of tanning and other materials into hides. In 1865 a patent was issued to William H. Towers describing a process of tanning or curing of hides in alcohol (1). Many investigators have studied solvent systems for

*Presented at the Fifty-third Annual Meeting, Lake Placid, New York, June 5, 1957.

the production of leather since that time (1–9, 11, 14–19, 21–24, 26, 27, 29–42). In 1953 the history and literature of solvent tannage was reviewed by O'Flaherty (34).

The author became interested in nonaqueous systems in 1935 during a study of the effects of solvents and solvent vapors on vegetable-tanned leathers (17). Several solvent-based tanning processes employing mineral, vegetable (21), and resin (24) tanning agents were developed before the outbreak of World War II. This work was resumed and expanded after the war. Processes for handling flat leather (40) as well as for producing molded leather shapes such as shoe uppers (33, 35, 38, 40) were developed and are commercially known as the Secotan and the Secomold processes, respectively. By 1950 a pilot machine and accessory equipment (36, 39) for processing pieces of hides 3′ x 4′ were designed and built. Operational constants obtained from this equipment were used to design the full-scale machine for the Secotan process. This machine (Fig. 3), built for processing side leather, was installed in the plant of the Eagle Ottawa Leather Company and was operated on an experimental basis for two years. In January, 1957, it was placed into regular production and has been in operation under production conditions since that time.

DEHYDRATION

The first operation in a nonaqueous system for manufacture of leather is usually dehydration. The term "dehydration" is used herein with reference to the removal of water from hydrophilic materials, such as hides, without causing shrinkage, shrinkage stresses, or adhesions. The best example of nearly perfect dehydration is freeze-drying. The material is quickly frozen, and the ice is sublimed from it under high vacuum. The hydrated structure s immobilized in the ice and remains dimensionally unchanged as the ice is sublimed from it until it is bone-dry. Freeze-drying is impractical for dehydration of hides or skins, although an ingenious method which overcomes most of the difficulties was devised by Cutler (25, 28) several years ago. Solvent dehydration accomplishes the same objective as freeze-drying and in proper equipment is inexpensive, rapid, and practical. Most solvents which are miscible with water may be used, one of the best being acetone.

Dehydrated raw hides from which the solvent has been dried have the feel and appearance of leather and are stable under ordinary atmospheric conditions except when they are wet with water. When immersed in water, they revert to their original condition. In order to prevent this reversion they must be tanned. Tanning materials may be introduced in the aqueous phase before dehydration or in the solvent phase after dehydration. If introduced in the aqueous phase, the presence of tanning materials assists in developing the desired leather characteristics in the finished product. If the tanning materials are introduced in the solvent phase after dehydration, their primary

effect will be to tan the hide without affecting its characteristics, which have been already established and fixed by dehydration. Such solvent tannages can result in excellent resistance to boiling water and to other conditions, but they contribute nothing to quality unless special steps are taken (41). Leather quality is governed by the feel, texture, temper, grain, and other such characteristics, but its stability and resistance toward water is taken for granted and is ordinarily judged to be satisfactory and not in need of improvement.

LEATHER QUALITY

The factor which governs the fundamental quality characteristics of the finished leather is the physical condition of the hide fiber structure at the time when it is immobilized through removal of water. When water is removed, the structure loses its plasticity and becomes self-supporting. The physical condition of the fiber structure depends to some extent on the total amount of water held, but particularly on its hydration and on the presence, or absence, of materials capable of combining with it. Hydration of raw hides can be varied over a wide range. The pickled hide is in the condition of least hydration, and the lime- or acid-swollen hide is at the other extreme. When a pickled skin on the one hand and an acid-swollen skin on the other hand are dehydrated in acetone, and the acetone is evaporated, two dry, leatherlike materials will be obtained. The fibrous structure in each will differ markedly from the other, and they will feel different and will have different textures. However, the differences are not caused by chemicals or tanning agents. The dehydrated, pickled skin is flat, thin, and parchment-like, but it is soft because there are no adhesions between the fibers. These characteristics of flatness and thinness were brought about by the low degree of hydration at the time of dehydration. If a portion of this dehydrated, pickled skin, still acetone-wet, is solvent-tanned in, let us say, a solution of a chromium salt in acetone, these characteristics will remain unchanged because the skin is nonplastic, although water has now become as inert a solvent toward it as acetone was toward the pickled skin. If a similar but water-wet pickled skin is chrome-tanned in the aqueous phase, using the same type of chromium salt, and is then dehydrated, its reactivity toward water is also eliminated, but in addition, the resultant leather is fuller, rounder, and of a texture which approaches more nearly that of good leather. The introduction of chromium in the aqueous phase modifies the plastic structure of the pickled skin, and this change accounts for the improvement. It must be emphasized that any other material producing the same physical change would serve equally well. If the material is not a tanning agent, then it will still be necessary to solvent-tan the skin after dehydration.

Examples of this type demonstrate that the quality of leather is established through control of the physical arrangement of the fiber bundles, such as

their spatial arrangement, and the amount of fiber contraction or relaxation existing at the time of dehydration. This fiber arrangement and condition depend in turn on the free water content and on the amount of tensioning or stress produced in the fiber structure by the hydration (bound water) and by the filling action of high-volume materials capable of combining with the hide substance, such as vegetable tanning materials, dyes, or resins, but not by inert fillers. The transition from this changeable structure to a fixed, set structure is accomplished when water is removed from the hides. In the solvent system this is accomplished by solvent dehydration. In leather made under aqueous conditions it is performed when the leather is air-dried for the first time.

In the conventional leathers made by aqueous processes, this setting of the structure occurs in the final stages of air-drying, when the amount of water remaining in the hides is insufficient to plasticize the structure and it, therefore, becomes immobilized, or set. The time of drying until this setting point is reached is relatively long, and as the water content in the hides is decreased through evaporation, changes in hydration and, therefore, in the structure of the still plastic fibers take place. If the drying is too fast, free water is removed faster than it can be regenerated by the fibers (syneresis), and coalescence of fibrils and fiber adhesions will occur. Physical forces applied at the time of drying, as when the hides are pasted or toggled, also contribute to the final characteristics by introducing stresses affecting the hide structure while it is still plastic. Internal shrinkages or dimensional changes caused by the diminishing water content further affect the results. Thus, the rate of drying is also a quality factor because it determines the time interval during which the structure can rearrange itself before immobilization occurs. Even the relative humidity at which drying is performed produces an effect and causes seasonal variations in some types of leather. The problem of keeping under scientific control this almost overwhelming number of variables is not easily solved. The greater portion of these variables is, however, eliminated when the transition from hydrated to set and fixed state is accomplished quickly, as for instance, by solvent dehydration. Compared to air-drying, solvent dehydration is practically instantaneous.

HIDE REACTIONS IN SOLVENT SYSTEMS

During the author's work with tanning agents in nonaqueous systems many interesting differences from their behavior in aqueous systems were observed. To summarize briefly several years of these investigations, it was found that the degree of fixation by hide substance is not so much a function of some intrinsic chemical characteristic of the material which is being fixed as it is a function of the vehicle in which the two are combined. The vehicle may be water, or organic solvents, or mixtures of liquids, or a gas. The degree of fixation by the hide substance of a given material will be a function of the

vehicle employed. No distinction is made here between fixation and tanning action, since the latter, like astringency, has a special meaning only with respect to water. The hide substance exhibits all the properties of a surface-active material, and fixation by it of dissolved substances follows more nearly the laws of adsorption than the laws of stoichiometric chemical reactions. The magnitude and the direction of the fixation reaction, that is, the adsorption or desorption of a compound by the hide substance, can be changed by the simple expedient of changing the solvent medium.

Thus, it was found that the fixation of acid from acetone is considerably greater than from an aqueous solution. If a hide which has combined with nearly its maximum amount of mineral acid in an aqueous system is dehydrated with acetone and is then brought in contact with more of the same acid dissolved in acetone, it will combine with an additional large quantity of acid until a new equilibrium is reached. When returned to water the acid will be given up by the hide until the original aqueous equilibrium is reestablished. Incidentally, if, after the fixation of acid from acetone, the hide is lightly washed in acetone to remove the uncombined excess without initiating desorption of the acid, and dried, a third equilibrium is obtained, since this changes the system from hide-acid-acetone to hide-acid-gas (air). If a volatile acid such as hydrochloric acid is used, much of it will be liberated by the hide and dissipated from it, provided the air is dry. The situation with a nonvolatile acid, such as sulfuric, is somewhat different because the system changes immediately from hide-acid-air to hide-acid-free acid, caused by the desorption of some acid which then remains at the interface. The conclusion drawn from these observations is that the acid-combining power of the hide substance for a given acid is primarily a function of the liquid or gas system in which the combination takes place.

The chrome-combining power of the hide substance is increased in an acetone system. An acetone-dehydrated acid skin will combine with, or adsorb, relatively huge amounts of zero basic chromium chloride. This chromium cannot be removed by repeated washings in acetone. However, if placed in running water, the reaction proceeds in the opposite direction, and the chromium is desorbed from the skin. If the water is acidified to maintain the over-all basicity of the skin during this washing at zero or less, desorption of chrome will be more complete. Neutral salts may be added to the water to inhibit swelling.

Data supporting the enhanced uptake of acid and of chromium by hide substance from nonaqueous systems will be presented in later communications.

The fixation of vegetable extracts is greatly reduced or entirely eliminated in nonaqueous systems. The author found that quebracho is not fixed by hide substance from solutions of dry acetone, methanol, or ethanol (21, 24). Lollar (23) also investigated the take-up or fixation of quebracho from acetone

and reached the same conclusions. The vigorous reaction between hide substance and quebracho in a water system is reversed in these solvents. Even when a thoroughly dried quebracho-tanned hide is placed into a methanol-water solution, the reaction proceeds in the opposite direction, and desorption of the quebracho takes place. The presence of water in methanol is helpful in this case to rehydrate the hide in order to, so to speak, unlock the material, but too much water will, of course, diminish or even inhibit desorption. Merrill and his coworkers (26, 27) reached significant conclusions from an extensive study which they made of the stripping of vegetable tannins from leather by aqueous organic solvents. An early process for solvent tanning with quebracho, developed by the author in 1939, consisted of impregnating a dehydrated hide with a solution of quebracho in a solvent from which it is not adsorbed by the hide substance, then drying the hide to remove the solvent, and then immersing it in water to set, or fix, the quebracho in the hide (21). An alternative method proposed by Roddy (22) and extended by Kremen (37) is to immerse a water-wet hide in an acetone solution of quebracho. Final fixation occurs in this case also through the action of water, because it is the last to leave the hide when the acetone-water mixture is dried from it. However, notwithstanding the fact that under ordinary circumstances there is no fixation of quebracho by the hide substance in nonaqueous systems, fixation can be developed without the use of water. For example, if a methanol solution of quebracho is alkalized (and maintained free of water), the dehydrated hide will combine with or adsorb the quebracho and exhaust it from the solution. Water is not needed to cause fixation of quebracho in this case, although it may be debated whether or not tanning occurs at this stage or when the hide becomes exposed to water at some later time. This behavior of quebracho is interesting because it demonstrates not only that the direction of the fixation reaction can be reversed by changing the solvent medium but also that the direction of the reaction in the same solvent may be reversed by a relatively minor modification of the system by the addition of a base.

The contention that hide fixation is physical in character and is reversible is supported also in the case of materials which appear to react more in a chemical manner with the hide substance. Formaldehyde may be taken as an example. A hide will combine with or fix more formaldehyde from an acetone solution than from water. When a dehydrated hide which has been treated with an acetone-formaldehyde solution is dried and immersed in water, the excess formaldehyde fixed by it is desorbed, and a new, lower, equilibrium is reached which is the same as it is for the fixation of formaldehyde from water. Pickard and his coworkers (9) found that formaldehyde will combine with hide substance in a gas system. In the dehydrated hide-gaseous formaldehyde–air-at-room-temperature system the hide appears to adsorb more formaldehyde than it does from water but less than from ace-

tone. This behavior of formaldehyde was investigated qualitatively and is presented here tentatively, subject to quantitative verification.

MECHANISM OF HIDE REACTIONS

This necessarily brief résumé of reactions of the hide substance in solvent systems simply augments the already imposing evidence gathered by many workers in the field of aqueous leather technology that its reactions are adsorptive in character and occur at the interfaces of its colloidal structure. Thus, the investigations of Elöd and Cantor (10, 13) and of McLaughlin and his coworkers (12, 20) on chrome tanning, of Chang and Doherty (18) on vegetable tannins, of Salcedo and Highberger (19) on formaldehyde, and of many other workers, tend to support this view. Some true stoichiometric chemical reactions undoubtedly do occur, but the preponderance of the reactions of importance to the production of leather are, in the opinion of the author, physical. The hide substance, according to this concept, is an extremely active adsorbent, and the fixation by it of an acid, or of a dye, or of a tanning agent is, in each case, the same phenomenon of adsorption. The quality differentiating the tannins from other substances which can combine just as vigorously is that the adsorption of the tanning material on the hide substance interfaces decreases the specific or preferential capacity of these interfaces to adsorb water and hence of the hide substance to become hydrated. Capacity to adsorb other materials may not be impaired and may actually be increased as, for instance, when the tanning material happens to be also a mordant. When the tanning is performed in the aqueous phase, the adsorbed water originally held becomes incompatible with the new tanning agent–collagen adsorption complex and is desorbed at the end of the tanning operation. It is, however, partially retained by pseudohydration, separating slowly from the hide substance by syneresis. Drying the tanned hide for the first time accomplishes the final removal of this water, which is irreversible under ordinary circumstances. Subsequent rewetting is substantially absorptive or capillary in character, and the degree of specific adsorption for water which still remains depends on the type and the amount of tannage received.

The study of the reactions of the hide substance in nonaqueous systems is informative and provides a useful research tool because similar investigations carried out in water are often obscured by side effects caused by its hydrophilic nature. In most solvents the hide substance behaves as a lyophobic material and is incapable of swelling or solvating. In this sense, an untanned, raw, dehydrated hide is already in a "tanned" state with respect to a solvent such as acetone. One of the most useful aspects of such studies is that the adsorptive capacity of hide substance for a given material can be varied by proper selection of solvent or solvent mixtures, as already described. Other applications include checking reactions which are suspected to be ionic in

character, since a proper choice of solvent can control the degree of ionization, and performing titrations where it is important to eliminate the by-products from the solution. The usual pH measurements are, of course, inapplicable and meaningless, but color indicators which have been calibrated in terms of the solvent being used are quite effective in determining the progress of a reaction. It is believed that the utilization and further extension and refinement of this approach may furnish clearer understanding in areas of leather technology where some degree of uncertainty still exists.

SECOTAN PROCESS

In the Secotan process the tanning materials may be applied either in the solvent or the aqueous phase. In the process as it is practiced at present they are applied in the aqueous phase. This situation may change when materials developed specially for solvent tannage become available in commercial quantities. At present there is little or no economic advantage in applying the more common tanning agents in a solvent system when making side upper leather. The reason for this is that most of the forms commonly used in tanneries are soluble with difficulty in solvents and the modifications which are soluble are usually more expensive. Conversely, there are definite advantages in using these materials in the aqueous phase when it is followed by dehydration. The most notable of these is the appreciable reduction in the amount of tanning material needed. For instance, in the case of chrome-tanned leather, less than a quarter of the normal amount of chromium is employed to achieve resistance to boiling water. Similar resistance to boiling water could probably also be achieved in regular aqueous leather manufacture with smaller quantities of chromium and more careful control, but the quality of the resulting leather would suffer. The quality of the leather produced by the Secotan process is not determined by the amounts of tanning agents employed but by the physical condition of the fibrous structure of the skins as they enter the solvent phase of the process, as already discussed earlier in this paper.

The process consists of two stages: the preparation of the hides for the Secotan machine, which is performed in water, and the processing of these hides through the machine in a solvent system. The basic operations required for making one type of side leather are shown in Fig. 1. Pickled sides, split in the lime, are loaded into a special drum. The liquors can be heated or cooled and pumped in or out of this drum while it rotates. The formaldehyde-salt solution shown in Box 1 is then fed to the drum, and the temperature is raised so as to reach 155° F. in 45 min. The liquor is then pumped from the drum into a storage tank where it is reconstituted to be used over again. The formulation shown in Box 2 is now fed into the drum. Note that the amount of chromium used is very low, being less than a quarter of the amount normally employed. After 45 min. running, the quebracho solution shown

in Box 3 is added, and 15 min. later the skins are washed. The total time for the pretreatment, excluding handling, is of the order of 2 hr.

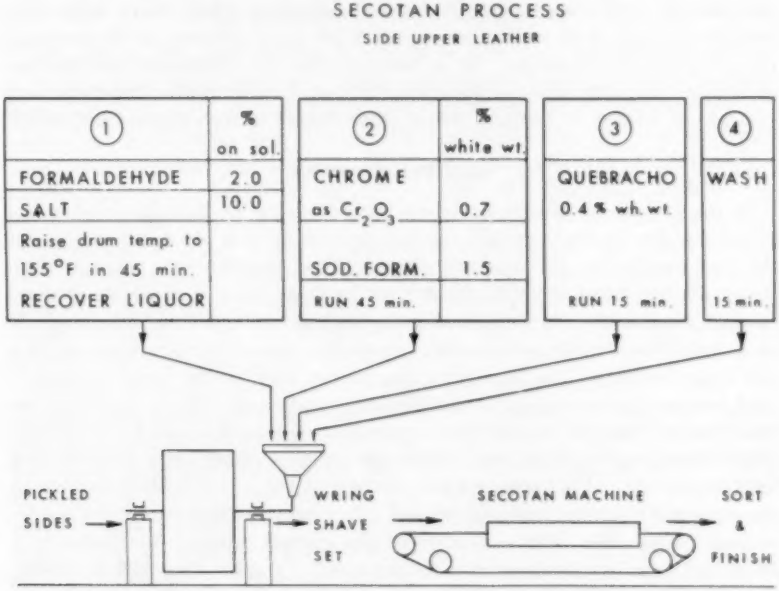

FIGURE 1.—Outline of Secotan process for making side leather.

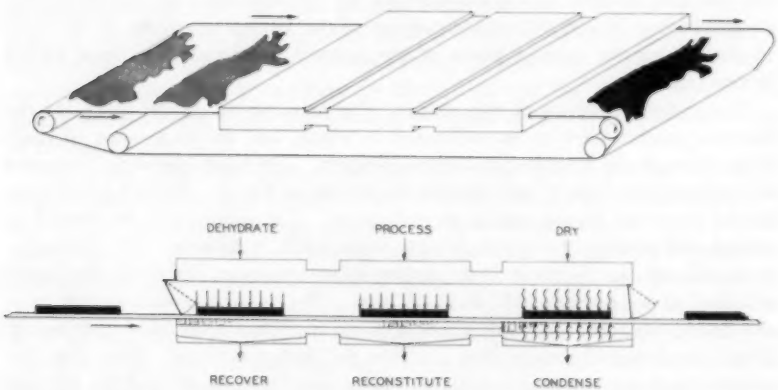

FIGURE 2.—Diagram of essential parts of the Secotan machine.

The pretreated skins are taken from the drum and are wrung, shaved, and set or put out in the usual manner. They are then ready for the Secotan machine in which they are dehydrated, fatliquored, and dried. After emerging from the machine the leather is ready for sorting and finishing. The machine will process up to 60 sides per hour.

A Secotan machine for side upper leather is approximately 13′ wide and 30′ long. An endless, smooth-surfaced, porous conveyor belt, 12′ wide, enters through automatically controlled shutters and passes through several interconnected, low-volume, totally enclosed chambers and emerges from the last chamber of the machine, also through a shutter system, to return to the starting point. Figure 2 shows the essential parts of the machine diagrammatically. An endless porous conveyor, on which the skins are slicked grain side down, is shown in the upper view as passing through the three chambers, or sections, of the machine and emerging on the right carrying the dry leather. Each section of the machine is structurally similar and includes an upper and a lower platen of a press, the surface of the lower platens being perforated. The platens are set approximately one inch apart, and the conveyor carrying the skins moves in the space between them. Each pair of platens is one section of the machine. The conveyor moves intermittently, carrying each skin successively into each section of the machine.

The lower view (Fig. 2) shows the machine in cross section with the vertical scale greatly exaggerated for clarity. The conveyor enters from the left carrying the slicked-out skins, shown as heavy black lines. The three sections are totally enclosed, and the narrow slots through which the conveyor enters and exits are closed by shutters which open automatically only when a skin passes underneath them. The total enclosed volume of the machine in which solvent vapors are present is small (roughly equivalent to a 3′ cube). The hides enter the machine water-wet and emerge dry. They are never handled while they are inside the machine.

At the first section of the machine the water-wet skin is dehydrated by forcing acetone through it. The acetone is delivered under pressure through suitable piping in the upper platen to the upper or flesh side of the skin and is forced through the skin, conveyor, and perforated lower platen. The effluent acetone, mixed with water from the skin, is pumped to the solvent recovery system, where the acetone is recovered and returned to storage. The dehydration time varies with skin thickness and the pressure used and ordinarily requires 20–45 sec. The conveyor carries the dehydrated skin, now wet with acetone, to the second section. Processing here may consist of forcing an acetone solution of fatliquor through the skin in a manner similar to the dehydration section, or it may involve tanning, dyeing, or the introduction of other materials into the skin to impart the desired characteristics to the finished leather. In some cases it is possible to combine several processing operations in one section. In other cases additional processing sections may

be required. Where side upper leather is processed as described in Fig. 1, processing in the second section consists of fatliquoring. The effluent fatliquor is automatically reconstituted and sent to stock storage for reuse.

In the third and last section of the machine the sandwich composed of the skin and conveyor is squeezed between two heated surfaces until the acetone is vaporized. This drying section is so arranged that the acetone vapor is carried away through openings in the lower platen and delivered to a condenser where it is condensed and recovered. The finished skin and conveyor, therefore, emerge dry from the machine. There is little chance of overheating the leather, since the vaporization of the acetone maintains the temperature at or below 135° F.

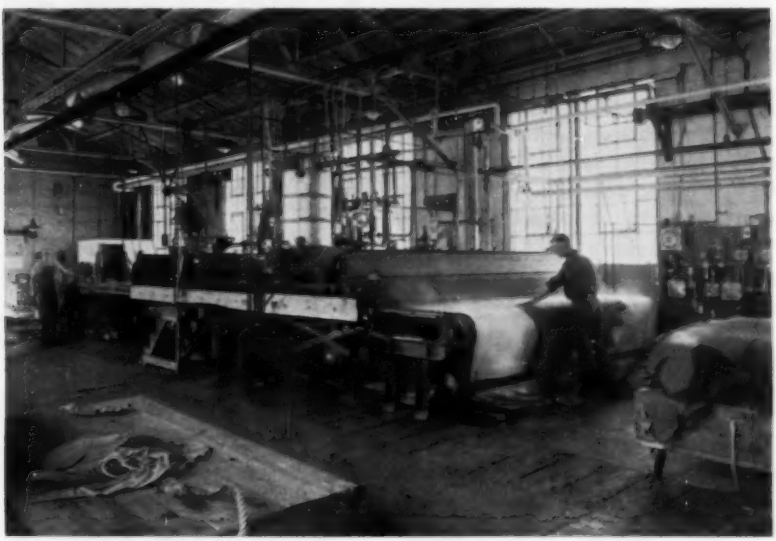

FIGURE 3.—General view of the Secotan machine.

In a three-section machine (Fig. 3) there will be three skins in process, and the cycle for the machine will be determined by the slowest of the three operations. This is usually the dehydration. If, as an example, the total time in the dehydration section is 1 min., then the skins remain in the machine for a total of 3 min. and must be fed to and removed from the machine at the rate of one skin per min.

Both the process and the machine are flexible. In setting the time for a cycle it is merely necessary to be sure that more than sufficient time is allowed for the slowest operation. It is impossible to overdehydrate or over-

process in any of the operations. Similarly, there is equal flexibility in the productive capacity of the machine. For example, if it were desired to shorten the 1-min. cycle in the example above, a second dehydration section could be added. The dehydration cycle would then be approximately halved to ½ min. each in the first and second dehydration sections, and the output of the machine would be substantially doubled, provided none of the other sections required more than ½ min. each. The ultimate limiting factor to the speed of operation is, therefore, not the equipment but the human factor: the speed of slicking the skins on the loading end of the machine.

The instrumentation is of the type usually found in automatic machinery and modern process industries, and consists essentially of sequential timers, automatic valving, and interlocking mechanisms. In addition, manual push-button control is provided for determining the proper cycling for new leathers. The equipment for reconstitution of effluent solvent liquors and for solvent recovery is similarly instrumented for fully automatic operation.

The conveyor consists of separate screen sections attached to and driven by roller chains on each side of the machine. The spacing between screens on the conveyor corresponds to the spacing between sections of the machine. Each screen is somewhat larger than an individual platen of a section. A chain indexing mechanism locates each screen section centrally in the stations. The surface of each screen is covered by a resin-impregnated, smooth, porous paper on which the skins are initially slicked out. This construction allows the individual sections of the conveyor screens or the papers covering the screens to be repaired or replaced without affecting the whole conveyor.

One of the essential mechanical features that allows the process to operate successfully is the sealing device which seals the irregular edges of each skin against the porous conveyor so that the solvent may be forced under pressure through the skins without spilling over the edges (36, 39). Figures 4–7 show the cross-sectional view of the dehydration station with the separation between the upper and lower platens again greatly exaggerated for clarity. Four successive stages of the sealing operation are shown.

The sealing device consists of a thin, rectangular, synthetic rubber diaphragm—or blanket, as it is called—of the same area as the upper platen of a section. This blanket is roughly 6′ wide and 12′ long for each section of a side machine. This blanket is shown in Fig. 4 as the wavy heavy line extending below the lower surface of the platen. The perimeter of this blanket is clamped to the upper platen in an airtight manner. An opening in the center of the blanket, approximately 6″ in diameter, is similarly clamped to the upper platen, in the center. Suitable piping is provided to admit compressed air to the space between the upper platen and the blanket as well as to the skin side of the blanket through the opening in the center which is also used to admit the acetone from a charge tank or reservoir.

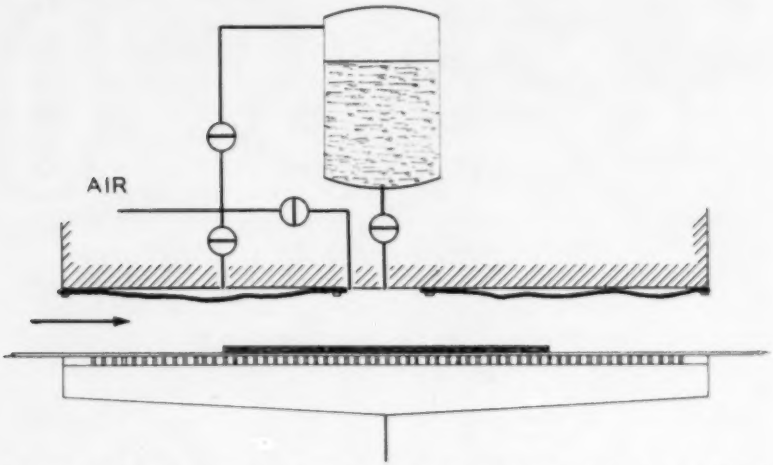

FIGURE 4.—Diagrammatic sectional view of the dehydration section. Blanket (heavy wavy line) in relaxed position.

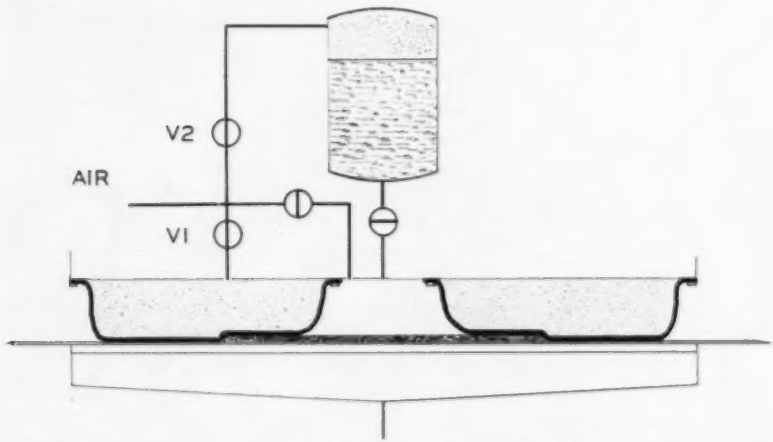

FIGURE 5.—Diagrammatic sectional view of the dehydration section. Blanket inflated to seal edges of skin.

Figure 4 shows the blanket in the relaxed position: the charge tank has been filled with the required amount of acetone for the dehydration operation, and the porous conveyor has just brought a skin (heavy black line on top of the lower platen) into position. All the valves are in the closed position at this instant.

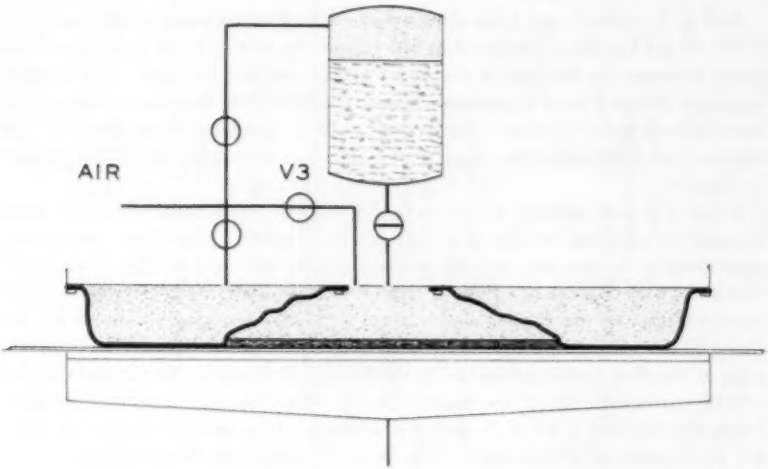

FIGURE 6.—Diagrammatic sectional view of the dehydration section. Air admitted to skin side of blanket.

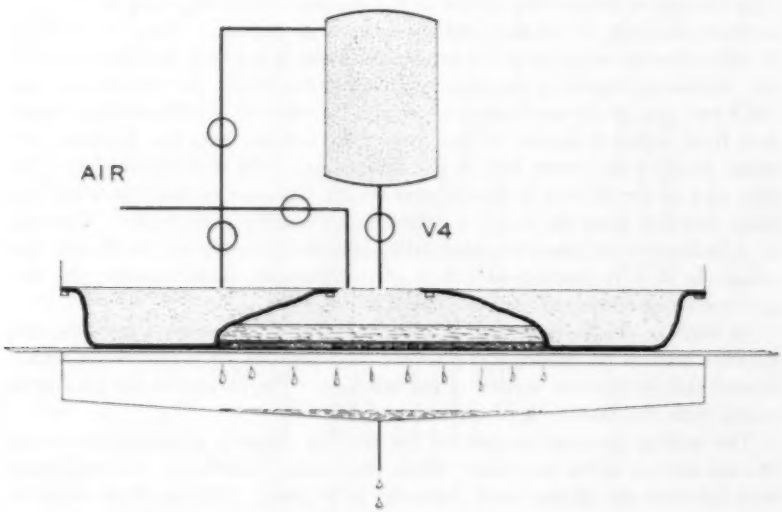

FIGURE 7.—Diagrammatic sectional view of the dehydration section. Acetone applied to flesh side of skin.

In Fig. 5, valves 1 and 2 have been opened, and compressed air at a pressure of 10–15 psi has been admitted to the top of the charge tank and also to the space between the bottom of the upper platen and the blanket. The blanket has been inflated until it presses against the conveyor and skin lying on the lower platen except in the center opposite the 6″ opening in the blanket. The blanket not only seals the edges of the skin but also seals any holes present in the skin.

Valve 3 is now opened, as shown in Fig. 6, and compressed air at the same pressure is admitted to the skin side of the blanket. The water-wet skin is impermeable to the air, and the pressure on either side of that part of the blanket which is over the skin is, therefore, equalized. The blanket portion over the area of the skin is therefore in a relaxed position as shown by the wavy lines. However, that part of the blanket which extends beyond the edge of the skin is still subjected to a differential pressure, the pressure acting on the conveyor side of the blanket being atmospheric. Since the pressure inside the blanket is 10 to 15 psi, this differential in pressure holds the blanket firmly around all the edges of the skin and causes an effective seal.

In Fig. 7, valve 4 has been opened, and the acetone which was contained in the tank has rapidly discharged and has spread in a uniform layer over the flesh side of the skin. Since the charge tank was at the same air pressure as the rest of the system, the acetone is delivered to the flesh side of the skin under the same pressure of 10–15 psi. It cannot spill over the edge of the skin because of the sealing action of the blanket at the edge and at the area between the edge of the skin and the edge of the section. Since the acetone is under pressure relative to the grain side of the skin which is in contact with the porous conveyor, it therefore permeates the skin, the conveyor, and the lower side of the perforated lower platen, where it is collected in a reservoir from which it is sent to recovery. The acetone acts like a piston in a sense, pushing the water held in the interstices of the skin ahead of it. The first part of the effluent is almost pure water. As soon as this first water has been removed from the skin, its permeability improves markedly. The rate of dehydration increases exponentially, and the dehydration is efficient because the skin is exposed to a flow of continuously fresh acetone, the wet acetone being constantly removed as it is formed.

As soon as the dehydration is complete, the air pressure is released, the blanket returns to its original position, and the conveyor moves the dehydrated skin to the next section of the machine. The total time for producing a seal with the blanket is of the order of 2 sec.

The sealing pressure exerted by the blanket requires a rigid construction of each section of the machine. When the blanket is inflated, the total pressure between the platens is of the order of 60 tons. Sealing of the skins in the processing section is exactly the same as it is in the dehydration section. In the dryer section of the machine (42) a similar blanket is used, but its construction is somewhat different.

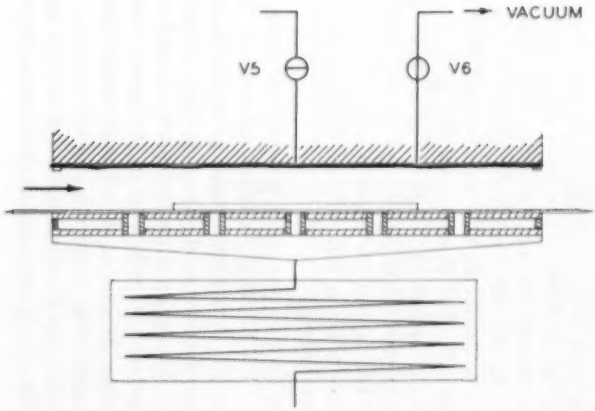

FIGURE 8.—Diagrammatic sectional view of the dryer section. Blanket under vacuum.

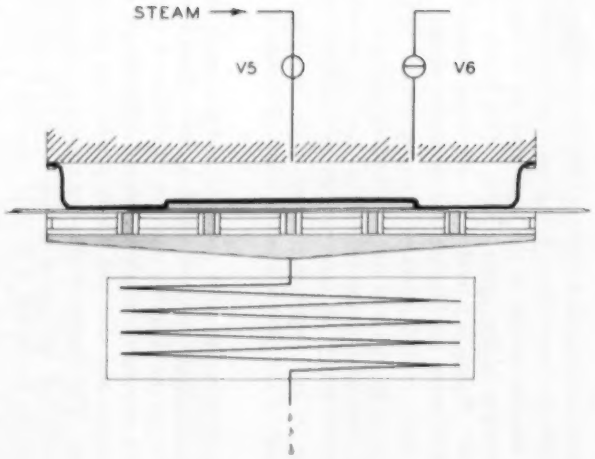

FIGURE 9.—Diagrammatic sectional view of the dryer section. Blanket under steam pressure.

Figures 8 and 9 show a cross-sectional view of the dryer section, the spacing between the upper and the lower platen being again exaggerated for clarity. The perimeter of a blanket similar to the type used for sealing is clamped tightly to the lower edges of the upper platen. This blanket contains no opening in the center. Piping is provided for admitting steam or applying vacuum to the space between the blanket and the upper platen. The lower platen resembles a steam-heated platen of a curing press. It is cored for steam which keeps it at an elevated temperature. In addition, a number of openings transversely through the thickness of the platen are provided. These openings are, of course, sealed off from the steam inside the platen, and they provide passage for any vapor originating at the lower surface of the skin to the bottom side of the platen where a manifold directs it into a condenser.

In Figure 8, valve 6 is in the open position, and the space between the platen and the blanket is under vacuum which keeps it from hanging down while the conveyor brings the skin into position. In Figure 9, the vacuum is closed off, and valve 5 is opened, admitting steam. This steam inflates the blanket against conveyor and skin and firmly presses the two against the heated bottom platen. The blanket is also hot, since it is inflated by steam. As the result of this intimate application of heat from both sides the acetone held by the skin (and the conveyor) is quickly vaporized. The pressure of the acetone vapor forces it from the skin and causes it to flow laterally between the blanket and the lower platen through the porous conveyor. Since the path through the openings provided in the lower platen offers less resistance than the lateral flow through the conveyor, the vapor quickly finds its way to these openings and to the vapor manifold located on the under side of the platen, from which it is conducted to a water-cooled condenser where it is condensed and recovered. The shaded area of Fig. 9 represents the path of the flow of the vapor through the openings in the lower platen. As soon as drying is completed, valve 5 is closed, and the vacuum is applied. The blanket is rapidly sucked against the upper platen, and the steam condensate which formed inside while it was inflated is forced out and is discharged through appropriate piping.

The leather made by the Secotan process is, generally speaking, softer and of lower apparent density. The physical properties are normal. The leather quality is markedly improved. Scar tissue, rough grain, brand marks, neck folds, and other surface imperfections tend to be upgraded. Figure 10 shows how this is accomplished. The upper view shows an enlarged section of a skin which contains exaggerated imperfections in the grain structure, such as scars or heavy neck folds. As soon as this skin enters the machine, it is subjected to fluid pressure, at a stage where it is still very plastic. The application of this uniform pressure has the effect of transposing the grain irregularities to the flesh side. The skin is subjected to this pressure in each section

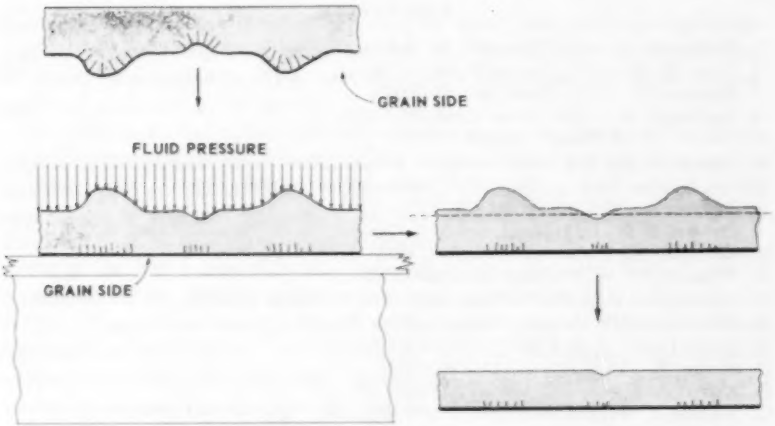

FIGURE 10.—How grain irregularities are minimized.

of the machine, including the last, where it is dried. This last operation irreversibly sets it in this transposed position. It emerges from the machine, therefore, with a flat, smooth grain which is permanently set. The looser areas of the hides are similarly benefited with a resultant increase in the cutting value. When the leather emerges from the machine, it is ready for sorting and finishing.

The process shortens the time of manufacture by some 8–12 days in the case of side upper leather. This, in turn, effects an appreciable inventory saving of stock-in-process. The process improves the uniformity of the leather by eliminating most of the variables from the more important leather-making operations. The leather produced has a better-defined fiber structure because of the lack of adhesions, and it is upgraded on an average of more than one grade because it is processed in the flat, slicked-out condition while in contact under pressure with the smooth surface of the conveyor.

ACKNOWLEDGEMENT

The author wishes to thank William C. Raftell, Susan Poulos, Jack A. Willard, Scott Berry, Mary Borg, and Alan S. Michaels, who worked with him in his laboratory during the investigation of solvent systems and the development of the process, as well as the staff of Artisan Metal Products, Inc., for their assistance and cooperation in the development of the experimental equipment. Last, but not least, he wishes to thank Sam B. Wardwell and his associates at Knowlton Brothers, Inc., for their tireless efforts culminating in the successful production of a porous material for the process.

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DISCUSSION

DR. T. C. THORSTENSEN (Lowell Technological Institute Research Foundation): It is so very seldom that leather manufacturers are faced with a truly revolutionary new process. When one does come along, they must

conscientiously sweep the cobwebs from their minds and readjust their thinking. Leather-making may be an ancient art, but it is not a sacred ritual. We must be prepared to make radical changes when and if it becomes practical and profitable to do so.

Mr. Ushakoff has taken solvent tannage from the realm of academic speculation to a production reality. The construction of the machine and the methods used should stimulate a good deal of thinking with regard to the application of new and different types of chemicals and to the possible production of entirely different types of leather.

There are two points that are pertinent to this question, I think, and I would like to use them in order to pose two questions to Mr. Ushakoff.

First, it is evident from the closed system that is being used that the solvent loss has been reduced to a very low level. I would like to ask Mr. Ushakoff to give us a little more specific information as to just what is the solvent loss in this system, in terms of the amount of leather produced.

Second, on the question of upgrading, just how great an advantage is given to this upgrading?

MR. USHAKOFF: Before I answer these questions, I would like to mention that the Eagle Ottawa Leather Company has been very kind in providing some leathers which are currently being produced by this process. These leathers are exhibited in front of the stage, if you wish to inspect them.

The solvent loss at present corresponds to about one-fifth of the solvent which is lost or used in finishing upholstery leather by spray-finishing. In terms of weight it amounts to a few ounces of acetone per side. I would rather not be more specific at this time because there are a number of known places where leakages occur in the present system, and they should first be eliminated. Notwithstanding this fact, the losses are very low.

We use a so-called one-stage dehydration, which means that the fresh acetone is used only once and then is sent to be recovered. In this method of dehydration as many stages of counterflow dehydration may be employed as are desired. If, for instance, three stages are used, the same amount of solvent, and therefore the same solvent loss, will produce three times as much leather. The solvent loss per square foot of leather produced will then be one-third of the loss with one stage. I can say that our losses are so low that we have never found it necessary even to consider using more than one stage for dehydration.

DR. THORSTENSEN: Now the question of upgrading?

MR. USHAKOFF: I would like to have Dr. Compton of Eagle Ottawa Leather Company answer that, if he will, please. He is much more qualified to answer it than I.

DR. ELL DEE COMPTON (Eagle Ottawa Leather Co.): I would like to comment on the acetone loss. The economics of the process in the inefficient system that Mr. Ushakoff refers to, is still favorable. The cost of materials, including the acetone lost, is less than the conventional materials.

As far as upgrading is concerned, it is the most important factor in the process. The sides that are on the table here for exhibit are all full-grain. They were not selected. The stock is not selected in the beamhouse. It is tannery-run—50 or 60 cows or steers. You may examine them for yourselves.

In comparing a few thousand matched sides in regular production, we found an average upgrading of 1¼ grades out of four. That does not include the upgrading into full grain.

DR. SEYMOUR S. KREMEN (Leather Research Corp.): I wonder if you will elaborate a bit on the uptake of quebracho in acetone on the alkaline side. What kind of substrate did you have? What do you mean by alkaline? How much water and alkali did you have with the quebracho and acetone?

MR. USHAKOFF: In initial tests we used anhydrous ammonia for the alkalizing of quebracho. This is a rather involved operation because as you begin to alkalize the quebracho solution its solubility decreases, and it is apt to precipitate. The quebracho-alakli complex which is formed is taken up by the dehydrated skin, and the solution becomes exhausted. However, complications arise because this complex is less resistant to water, and the skin is not well tanned with respect to water until this complex is converted to a more stable form.

DR. KREMEN: In what condition was the stock before you started this alkalizing?

MR. USHAKOFF: It was a dehydrated, depickled skin.

DR. KREMEN: If you had a bated hide which had a minimum deliming and was therefore largely alkaline and had been dehydrated, would you expect a large amount of quebracho in acetone to be taken up?

MR. USHAKOFF: I do not know. Too much alkali in the hide may cause precipitation.

DR. H. B. MERRILL (B. D. Eisendrath Tanning Co.): As I understood the presentation, the beamhouse processes were carried out by conventional methods?

MR. USHAKOFF: Yes.

DR. MERRILL: And the stock was pickled?

MR. USHAKOFF: Yes.

DR. MERRILL: Then it was given a tannage in an aqueous system, first a formaldehyde tannage and then a chrome tannage, which might or might not be followed by a vegetable tannage?

MR. USHAKOFF: Correct.

DR. MERRILL: All using very small amounts of tanning materials and very short tanning time?

MR. USHAKOFF: Yes.

DR. MERRILL: It would seem then that the methods of tanning must have been modified considerably from the conventional methods in order to get that tannage with those small amounts of materials in a very short time. Will you elaborate on that?

MR. USHAKOFF: The formulation and the method are exactly as shown. The use of small amounts of materials is made possible by the fact that these skins are then run through the machine where they are processed in the nonaqueous phase. The heating cycle during the pretreatment contributes to the short time required to introduce these materials into the skins.

DR. MERRILL: Do you carry out your tannage in a very low float?

MR. USHAKOFF: Yes, we prefer to use as low a float as is practical.

DR. MERRILL: It is generally supposed that a pretreatment with formaldehyde, no matter how the leather is tanned afterwards, results in a somewhat thin leather rather than a plump leather. Have you observed that?

MR. USHAKOFF: No, I would not say so. The usual formaldehyde tannage is conducted at a higher pH than we employ. The heating cycle during the formaldehyde pretreatment is quite essential, and these two factors, plus the solvent operations which follow, modify the result.

IMPREGNATION OF SOLE LEATHER WITH POLYMERS*†

BERNARD H. FOUQUET AND JOSEPH R. KANAGY

National Bureau of Standards
Washington, D.C.

ABSTRACT

The utilization of elastomers and elastomer-resin blends for upgrading sole leather now practiced to a limited extent in the industry has prompted the determination of some of the important principles involved and a comparative study of properties imparted to the leather by different materials.

Evaluations made with elastomers such as Butyl rubber, polybutene, and polyisobutylene blended with a polymerized resin, and with a thermoplastic resin plasticized with oils indicate that the molecular weight of the impregnant is a primary consideration for proper impregnation of unfilled leather. Elastomers having molecular weights over 60,000 are unsatisfactory. Best results with ease of handling are obtained for elastomers such as polybutene and polyisobutylene with molecular weight no greater than 10,000. Improvements in water resistance and abrasion resistance are slightly greater per unit of impregnant for Butyl rubber than for the polybutenes. The improvements in these two physical properties are, in general, proportional to the amounts of the impregnants deposited in the leather.

Wear tests with vegetable-tanned sole leather indicated improvements in the order of 50% for the elastomer-resin treatment and lower-order improvements for resin–castor oil treatments.

—x—x—

INTRODUCTION

Previous research at the National Bureau of Standards has indicated the possibilities of using Hevea and Castilloa rubbers (1), polyisobutylene (2), and other polymers (3) as impregnants for upgrading and improving the wear of sole leather. Efforts to upgrade sole leather are concentrated primarily on the improvement of serviceability with respect to resistance to

*This study was sponsored by the Bureau of Supplies and Accounts of the Department of the Navy as part of research and development project NT 001-018.
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abrasion and water absorption; the objective is to find impregnants that achieve this purpose without imparting to the leather properties that are undesirable. In addition, the ease with which the impregnant can be dissolved is an important factor. The solutions should have a low viscosity at concentrations high enough to give adequate uptake in the leather.

Blends of Butyl rubber, polyisobutylene (Vistanex), or polybutene (Oronite) with a polymerized resin (Polypale), and mixtures of a thermoplastic resin (Vinsol) with tall oil and castor oil are evaluated in this paper as impregnants for sole leather.

The blending of a resin with an elastomer for impregnating leather has several advantages. An elastomer such as Butyl rubber, for example, is not a satisfactory impregnant for leather when used alone. In Varsol* the maximum concentration obtainable is 20% which results in a viscous solution (29 poises) that will give an uptake of only 6% in crust leather. By blending Butyl rubber with Polypale resin in Varsol it is possible to decrease the viscosity of the solution and obtain an increase in the amount of deposition. Polypale resin has the advantage of being very soluble in Varsol and of stiffening the leather to counteract the effect of the elastomers.

MATERIALS

The Butyl rubber† and Vistanex‡ used in this work were obtained from the Standard Oil Company of New Jersey. Oronite** was obtained from the Oronite Chemical Company. The Vinsol†† and Polypale‡‡ resins were obtained from the Hercules Powder Company.

A distilled grade of tall*** oil was used and the castor oil was a No. 1 C. P. grade.

Natural crust (unfilled and unrolled vegetable-tanned) sole leather was generally used for impregnation.

PROCEDURES

All elastomers or mixtures of elastomers and resins were applied to leather in solutions. The plasticized Vinsol was applied in isopropyl alcohol solutions, whereas the other polymers were used in Varsol solutions. Isopropyl alcohol was used for Vinsol, since it is insoluble in hydrocarbon solvents.

Impregnation of the leather consisted simply of immersing the unfilled sample in the solutions for a period of 2–3 hours, wiping off the excess, and

*Varsol is the trade name of the Standard Oil Company of New Jersey for a mixture of hydrocarbons boiling at 150°–200° C.

†Butyl rubber is a trade name for a copolymer of isobutylene and isoprene, also known as GR-I when it was produced in Government-owned plants.

‡Vistanex is a trade name for polyisobutylene.

**Oronite is a trade name for a low-molecular-weight polybutene.

††Vinsol is a dark-colored, thermoplastic resin composed of a complex mixture of various chemical components derived from southern pinewood.

‡‡Polypale is a light-colored resin prepared by the polymerization of rosin.

***Tall oil consists of a mixture of rosin acids, fatty acid sterols, and unsaponifiable matter obtained from the waste liquors of wood pulp manufacture.

evaporating the solvent at 40°–50° C. in a forced-draft oven to constant weight. The percent deposition is calculated from the increase in dry weight after impregnation and is based on the original weight of the leather dried under the same conditions. The concentrations of the solutions are given as percentage of solids on a weight basis.

The impregnated leathers were evaluated by water absorption and abrasion tests in the laboratory and by regular wear tests with shoe soles. Wherever needed, comparisons were made with finished vegetable-tanned leather taken as nearly as possible from corresponding areas on the hide. The tests on abrasive resistance and water absorption were made in accordance with methods in Federal Specification KKL-311a.

PROPORTION OF IMPREGNANTS IN BLENDS

In determining the ratio of elastomer to resin in the impregnating solutions which imparts the highest wear resistance, abrasion tests were performed on specimens impregnated in solutions in which the concentration ratios were varied. Some of these results are given in Table I. Results with

TABLE I

ABRASION RESISTANCE OF LEATHERS TREATED WITH DIFFERENT PROPORTIONS OF ELASTOMERS AND RESINS

Blend Ratio (by weight)		Concentration %	Deposition %	Thickness Lost on Abrasion per 12,000 Revolutions (mils)
Controls (finished veg.-tanned)			0	134
Butyl	Polypale			
60	25	24.3	7.6	118
50	50	28.6	10.9	105
40	75	32.9	14.3	105
30	100	37.2	18.8	105
20	125	41.4	21.8	119
10	150	45.7	29.2	125
Oronite	Polypale			
2	3	50	20.4	106
1	2	50	19.1	108
1	1	50	19.8	132
Tall oil	Vinsol			
1	1	50	26.0	114
1	2	50	27.0	109
3	5	50	26.0	115
Castor oil	Vinsol			
1	1	50	25.0	97
1	2	50	26.0	112
3	5	50	28.0	111

Butyl rubber and Polypale resin in which the ratios of Butyl to Polypale were varied from 12:5 to 1:15 indicate that as the proportion of resin is increased the uptake by the leather increases. However, maximum abrasion resistance is obtained only in the range of ratios 1:1 to 3:10. Leather treated in solutions containing elastomer and resin in the ratios of 1:3 to 1:4 is too stiff and hard. The proportion of elastomer to resin selected for use in the further tests was 2:3 . Results given in Table I indicate that this proportion is also satisfactory for Oronite and Polypale. The proportion chosen for the oil and resin mixtures was 3:5.

EFFECT OF VISCOSITY, MOLECULAR WEIGHT, AND SOLUTION CONCENTRATION ON IMPREGNATION

A study of the relation between characteristics of the impregnation solutions and impregnation of unfilled leather was made. Some of the results are shown in Table II. To determine the influence of solution viscosity on

TABLE II

INFLUENCE OF CONCENTRATION AND VISCOSITY ON DEPOSITION OF IMPREGNANTS IN CRUST LEATHER

Impregnant	Molecular Wt. (Approx.)	Temp. ° C.	Concentration %	Viscosity (poises)	Deposition %
Polypale* with:					
Butyl 268	56,000	25	38	129	10.2
"	"	25	25	—	6.3
"	"	40	38	68	9.7
Butyl 035	35,000	25	38	42	12.7
"	"	25	29	9.7	10.1
"	"	25	19	1.6	9.0
"	"	40	38	27	11.8
"	"	40	19	1	5.6
Vistanex LM-MS	8,700–10,000	25	38	0.86	19.5
Vistanex 80	64,000–81,000	25	38	350	10.6
Oronite 128	1500	25	75	18.5	37.0
"	"	25	60	—	31.0
"	"	25	50	0.93	23.0
"	"	25	38	0.12	19.0
"	"	25	25	—	13.0
"	"	25	12.5	—	7.0
Oronite 32	1200	25	38	0.09	20.0
Vinsol† with:					
Castor oil	—	25	50	—	29.0
"	—	25	38	—	22.0
"	—	25	25	—	15.0

*Ratio .. 2 parts elastomer: 3 parts Polypale resin.
†Ratio .. 3 parts castor oil: 5 parts Vinsol.

deposition, solutions containing Butyl 268 and 035 were tested at 25° C. and 40° C. Since the two Butyl rubbers have different molecular weights, they produce solutions that have different viscosities at similar concentrations. Viscosity is highly dependent on temperature and therefore may be varied simply by using different temperatures. The results show that although the viscosities are only about one-half as great at 40° C. as at 25° C., the amount of impregnant deposited in the leather is slightly less at the higher temperature. The amounts of Butyl 035 deposited at both temperatures in the leather are slightly greater than the amounts of Butyl 268. The latter has the higher average molecular weight.

It appears from these results that molecular weight rather than viscosity determines the amount of deposition. This is shown not only for the Butyl rubbers but also for the Vistanex elastomers. The Oronites, which have comparatively low molecular weights, show high depositions. High depositions are also shown with the Vinsol and oil formulations where the exact molecular weights are not known but are known to be low. Although the study is far from exhaustive with regard to molecular weight because the samples were not selected from all molecular weight ranges, it may be stated definitely that any elastomer over 60,000 M. W. is not suitable. Elastomers from about 15,000 to 60,000 M. W. may be used with difficulty, and theoptimum molecular weight range for ease of handling and best likelihood of success is 1,000 to 10,000.

TABLE III

RELATION OF PERCENTAGE DEPOSITION TO WATER ABSORBED

Impregnant	Deposition %	Water Absorbed in 24-hr. Period %
Control	—	55.9
Polypale* with:		
Butyl 035	12.7	37.4
"	10.1	43.6
"	9.0	46.2
Butyl 268	10.2	52.1
Vistanex LM-MS	19.5	41.5
Vistanex 80	10.6	51.6
Oronite 128	23.0	32.0
"	19.0	37.4
"	13.0	43.8
"	7.0	51.1
Oronite 32	20.0	38.2
Vinsol† with:		
Castor oil	29.0	28.0
Tall oil	27.0	36.0

*Ratio .. 2 parts elastomer: 3 parts Polypale resin.
†Ratio .. 3 parts oil: 5 parts Vinsol.

It is expected that the amount of a particular material deposited should be related to the solution concentration. Depositions in the leather for the Oronites and for the Vistanex LM-MS are almost exactly one-half the solution concentrations. The elastomers that have molecular weights higher than the Vistanex LM-MS give depositions much less than one-half the solution concentration. Vinsol gives depositions slightly greater than one-half the solution concentration.

EFFECT OF IMPREGNANTS ON WATER ABSORPTION

One of the most important improvements in properties brought about by the impregnation process is the reduction in the water absorption of the

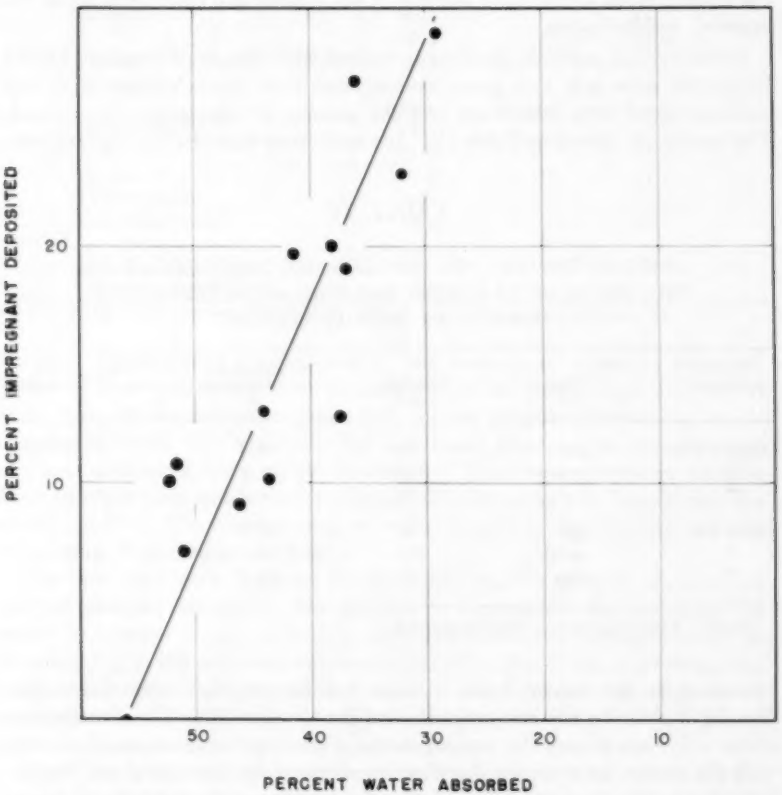

FIGURE 1.—Influence of the amount of impregnant deposited on the water absorption of leather.

treated leathers. Data on the water absorption obtained by immersion for 24 hours of the impregnated leathers are given in Fig. 1 and Table III. The results in Fig. 1 indicate that the reduction in water takeup is, in general, directly proportional to the amount of impregnant in the leather. Close examination of the data given in Table III indicates that extremely low reduction in water absorption per unit deposition occurs with Butyl 268 and Vistanex 80, which are elastomers of high molecular weight. The greatest reductions are obtained with treatments of Oronite and Vinsol, which are deposited in large percentages. These materials of low molecular weight probably give a more uniform deposition throughout the thickness of the leather, whereas those of high molecular weight are deposited on or near the surface. The latter type of deposition does not prevent water from entering the smaller pores in the outer portion of the leather and concentrating in the internal, unfilled pores.

To study this principle, specimens treated with blends of Oronite 128 and Butyl 268 were split into grain, corium, and flesh layers. Each layer was then extracted with chloroform and the amount of impregnant determined. The results are shown in Table IV. It can be seen that the impregnant con-

TABLE IV

DISTRIBUTION OF THE IMPREGNANT THROUGHOUT THE THICKNESS OF LEATHERS TREATED WITH DIFFERENT MOLECULAR WEIGHT BLENDS

Impregnant*	Split	Thickness (mils)	Concentration of Impregnant %	Concentration per Mil Thickness %
Oronite 128	grain	50	50.4	1.01
"	corium	132	14.0	0.11
"	flesh	76	27.2	0.36
Butyl 268	grain	45	14.8	0.33
"	corium	125	2.7	0.02
"	flesh	87	12.8	0.15

*Ratio .. 2 parts elastomer: 3 parts Polypale resin.

centration in the corium layer is much less for the high-molecular-weight Butyl 268 than for the low-molecular-weight Oronite 128. The distribution of the polymers among the various strata of the leather agrees qualitatively with the results for pore-size distribution obtained by Stromberg and Swerdlow (4) who list the pore sizes as being the greatest in the grain layer, intermediate in the flesh layer, and smallest in the corium layer.

EFFECT OF IMPREGNANTS ON ABRASION RESISTANCE

The abrasion resistance of leathers impregnated with each of the blends is given in Table V. The data as plotted in Fig. 2 indicate that the increase in

TABLE V

INFLUENCE OF POLYMER DEPOSITION ON ABRASIVE RESISTANCE

Treatment	Deposition %	Improvement in Abrasion Resistance %
Butyl 035	12.7	24.5
"	20.4	38.8
Butyl 268	10.2	16.5
"	19.1	32.9
Vistanex LM-MS	19.5	37.9
Vistanex 80	10.6	13.3
"	16.6	15.1
Oronite 128	19.0	29.2
"	31.0	57.0
Oronite 32	20.0	31.0
Tall oil–Vinsol	26.0	16.6
Castor oil–Vinsol	25.0	20.7

Ratio . . 2 parts elastomer: 3 parts Polypale resin
3 parts oil: 5 parts Vinsol

Where two values are given for one impregnant, the first was obtained by a single treatment and the second was obtained by several successive treatments as needed to raise the deposition to about 20%.

abrasion resistance is proportional to the amount of material deposited. Three or four treatments were necessary with the Butyls and Vistanex 80 to increase the deposition to about 20% for the purpose of obtaining results comparable with the Oronites. The first result given in the table for each of these rubbers is for a single treatment. The improvement in abrasive resistance per unit amount of impregnant is highest for the Butyls and Vistanex LM-MS. The improvement with the Oronites is slightly lower per unit of material deposited in the leather.

The two tests with Vistanex 80 show low improvement in abrasion per unit of material deposited. An attempt to increase the deposition of Vistanex 80 brought it only to 16.6% after four successive impregnations. As shown in Fig. 2, the improvement was not equal to that of the other elastomer-resin treatments. It is assumed that most of this elastomer was concentrated on the two surfaces and was not distributed properly for effective improvement on account of its high molecular weight.

The Vinsol-oil treatments, as shown in Fig. 2, give lower values for improvement in abrasion resistance in comparison to those produced by the elastomer-resin blend treatments.

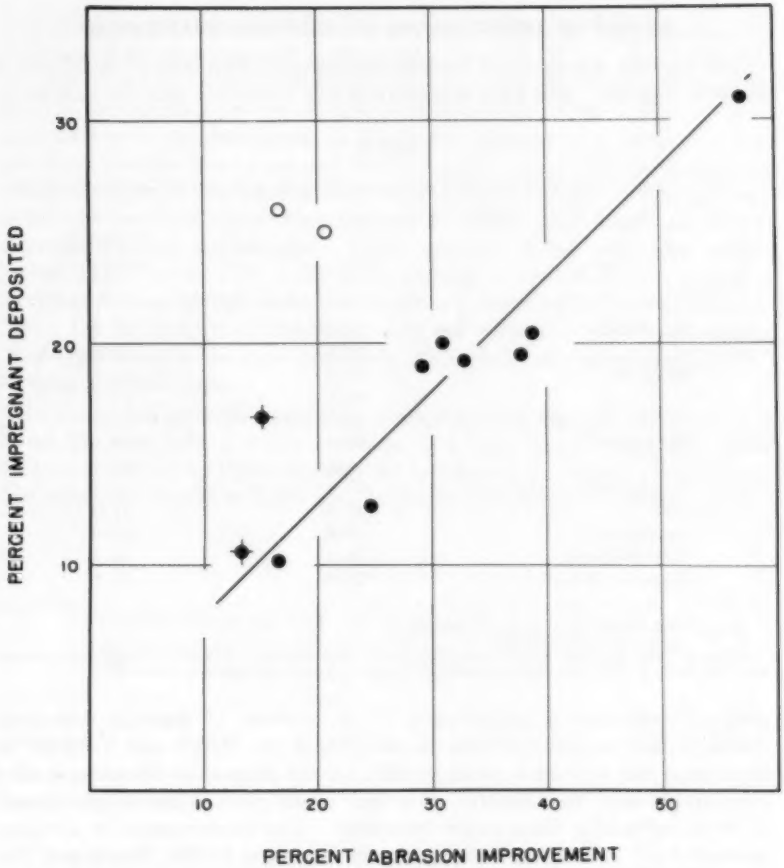

FIGURE 2.—Influence of the amount of impregnant deposited on the improvement in resistance to abrasion. Leathers impregnated with:
● Elastomer-resin blends
⊕ Vistanex 80 blend
○ Vinsol-oil

EFFECT OF IMPREGNANTS ON WEAR

Wear tests were made to evaluate the performance of Vistanex, Butyl rubber–Polypale, Oronite 128–Polypale, and Vinsol plasticized with castor oil. In the Vistanex and Butyl rubber–Polypale treatments, half of the impregnated soles were matched with regular vegetable-tanned leather and half with controls oil-dipped according to Navy regulations (5). The controls for the Vinsol and Oronite 128–Polypale were all oil-dipped.

The tests were stopped at the first sign of "break through" or hole in one of the soles. The wear of the soles was obtained from measurements taken at nine template positions before and after wear (6).

Improvement in wear due to impregnation was calculated as follows:

$$\text{Time for treated soles to wear 100 mils: } \frac{t \times 100}{W} = K$$

$$\text{Time for control soles to wear 100 mils: } \frac{t \times 100}{w} = k$$

where W and w are wear in mils in time t of treated soles and control soles, respectively.

$$\text{Percent improvement: } \frac{K - k}{k} 100 = \left(\frac{w}{W} - 1\right) 100$$

The improvements given by the various treatments were calculated using vegetable-tanned sole leather as a basis of comparison. The results are shown in Table VI.

TABLE VI

IMPROVEMENT IN WEAR OF SOLE LEATHER IMPREGNATED WITH DIFFERENT ELASTOMERS AND RESINS

Treatments	Type of Leather Impregnated	Pairs of Shoes	Deposition %	Improvement %
Vistanex	Vegetable-tanned	22	15–19	55
Oronite-Polypale	"	7	20–23	51
Butyl-Polypale	"	24	16–20	60
Vinsol–castor oil	"	7	20–25	31
Oil, sole treating, Military Spec. Mil-C-1829	"	63	15–18	18
Vinsol–castor oil	Chrome-retanned	7	24–26	70

A test performed on chrome-retanned soles treated with Vinsol and castor oil showed an improvement of 70% whereas vegetable-tanned leather treated with the same impregnant showed only 31% improvement. The difference in improvement is an indication of the better performance of chrome-retanned leather as has been demonstrated in previous wear tests (7).

TREATMENT OF FINISHED LEATHER

Tanners have shown an interest in the application of impregnants to finished sole leather. A few experiments were made to determine the results obtained in such treatments. Results on the impregnation of finished sole leather in Oronite-Polypale (2:3) solutions having different concentrations are shown in Table VII.

TABLE VII

DEPOSITION OF ORONITE 128–POLYPALE IN FINISHED VEGETABLE-TANNED SOLE LEATHER

Solution Concentration %	Deposition %
75	14.8
50	13.1
38	12.3

Where unfilled leather takes up 37, 23, and 19%, respectively, for the same solution concentrations, finished leather takes up amounts which are not greatly different for each of the different concentrations and are certainly not high enough to produce any substantial improvement in wear resistance. At the maximum deposition, the resulting improvement in abrasion resistance is only about 10%. It, therefore, does not appear feasible to impregnate finished leather.

CONCLUSION

From the results of this investigation it is indicated that the molecular weight of the elastomer should be a prime consideration in the choice of an impregnant. A low-molecular-weight elastomer such as Vistanex LM-MS or Oronite 128 can be made easily into a low-viscosity solution of high enough solids content to deposit an effective quantity of impregnant into the leather. With the materials described, improvements of the order of 50% in wear resistance and 35% reduction in water absorption can be expected from a deposition of 20% of the impregnant in the leather.

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2. ———, J. J. Davis, and R. A. Kinmonth, *JALCA*, **50**, 16 (1955).
3. ———, and T. J. Kilduff. *JALCA*, **44**, 151 (1949).
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5. Military Specification, Compound, Oil, Sole Treating, Mil-C-1829, December 9, 1949.
6. Oehler, R., S. Dahl, and T. J. Kilduff. *JALCA*, **47**, 642 (1952).
7. Bowker, R. C., and W. E. Emley. *JALCA*, **30**, 572 (1935).

Received March 28, 1958.

DISCUSSION

J. F. Wagoner (Armour Leather Company): This matter of upgrading the wearing quality of sole leather by impregnating with various polymers is one that is receiving a great deal of attention at the manufacturing level today. The Bureau of Standards has contributed greatly to our knowledge of this treatment over the past several years and again today has given us further pertinent data which I am sure will be helpful to all of us.

There is a strong feeling in some quarters that this resin-impregnated type of leather is going to go a long way toward helping the industry to regain some of the business lost to the synthetic and substitute soling materials.

I would like to ask Mr. Fouquet two questions: In your work to date, have you found that soles from the looser-textured belly and break areas show a larger percentage increase in wearing quality than soles from the backbone area? In other words, does the impregnation tend to level off the wearing quality of the bend as a whole?

Mr. Fouquet: So far, of course, we have not conducted any tests to show definitely the results of position. However, I can say this: Since the looser portions of the belly seem to take up the impregnant to a much greater extent, it would be expected that they would give about the same value in wear.

Mr. Wagoner: Would you expect them to level off the wearing qualities?

Mr. Fouquet: Yes.

Mr. Wagoner: In the actual wear test that you showed, we noted that you found with the Oronite-Polypale mixture an improvement of approximately 50%, whereas with the Butyl rubber–Polypale mixture the percentage improvement was 60%. Do you think it would be feasible to make a mixture composed of Butyl rubber, Oronite, and Polypale, not only to give further improved wear but also to add some flexibility? And if that is so, what proportions would you suggest?

Mr. Fouquet: I imagine from the results that I presented, showing the difference in distribution of these elastomers in the leather, it may prove advantageous to use a mixture of the two. However, I do not believe that the difference between 51% given by the Oronite and 60% given by the Butyl rubber would warrant the use of the mixture, because they would seem to be approximately the same.

Dr. Boris Monsaroff (Hamilton, Ontario, Canada): I would like to hear about the technique of impregnation. Did you dip the leather in the solution of the resin and then let it dry by evaporation, or did you pass the leather through rollers?

MR. FOUQUET: We just dipped the leather in the impregnating solutions and then, after wiping off the excess from the surface, let it dry.

STEPHEN A. SHIVAS (John A. Lang & Sons, Ltd.): How many pairs of shoes did you use for each of these treatments in the practical wear test?

MR. FOUQUET: We used about 20 for the lowest number and about 30 to 35 for the highest number.

MR. SHIVAS: Do you think that is sufficient to give a practical, significant result?

MR. FOUQUET: Well, there had been some statistical analyses done on previous wear tests where we used from 20 to 50 pairs. These analyses showed that the results were significant. So we continued using that number.

MR. SHIVAS: Have you done work comparing impregnated soles with any of the composition soles such as Neolite?

MR. FOUQUET: No.

W. R. KENZIE (Ontario Research Foundation): I would like to ask, first, whether I understand correctly that you came to the conclusion that the viscosity was not a governing factor in degree of deposition and therefore that a temperature above that of room temperature was not used; and auxiliary to that, I would like to ask what impregnation period was used.

MR. FOUQUET: In answer to your first question, you are right. We found that the viscosity of the solution seemed to be no governing factor in the deposition. The molecular weight of the elastomer was the governing item. So there was no point in reducing the viscosity of the solution by increasing the temperature. The immersion period was carried on from two to three hours.

MR. KENZIE: In other words, in the period of two to three hours you got the same uptake at room temperature as at 40° C.?

MR. FOUQUET: The final uptake was the same. I am not saying the rate was the same. The rate might have been different. But that we did not check.

MR. KENZIE: My other question is whether you found good correlation between the wear test figures and the abrasion test figures.

MR. FOUQUET: Actually, we have made no attempt to find any correlation up to this date. However, as I pointed out, in the abrasion tests the resistance of the Butyl was the highest, and of course the same results were given in the wear test. The Vistanex came next, and after that Oronite 128 and the Vinsol.

MR. KENZIE: You got, I take it, a higher percentage of improvement in the wear test than in the abrasion test. Is that correct?

MR. FOUQUET: You mean the magnitude of the improvement was different?

MR. KENZIE: Yes.

MR. FOUQUET: Yes, but there the results were calculated differently. In the wear test the results were calculated as the improvement in wear life, whereas in the abrasion tests the percentage improvement over the control was calculated, and that is all.

R. G. ASHCRAFT (Endicott Johnson Corp.): What finish was used on the leather after immersion?

MR. FOUQUET: No finish was used.

MR. ASHCRAFT: Will you define for us just what you consider crust leather to be? At what point was that taken?

MR. FOUQUET: What I consider to be crust leather in these tests is unfilled leather, whereas filled vegetable-tanned leather would be leather containing salts and glucose—in other words, water solubles.

MR. ASHCRAFT: Did this crust leather have any application of oil of any kind?

MR. KANAGY: I would like to answer that. The crust leather that we used was obtained from a tannery and was processed to the point just before it goes into the oil wheel. It was treated with a small amount of sulfonated oil. Another thing I would like to say is that at the Bureau we do not do any work on finishing of these soles. We have no facilities for working on finishing, and I think we are going to leave it up to the tanners to work out their own finishing processes.

A. N. KAY (Howes Leather Co., Inc.): You people have improved one property of the leather. What about the other properties of the leather—the water vapor permeability, puncture resistance, and so on? Have those been examined?

MR. FOUQUET: As for the other properties of leather, the only one that was tested was the dimensional stability. It seemed to decrease a little bit with impregnation. But for extensive analysis, abrasion resistance and water absorption were the only properties tested.

MR. KAY: Are you planning to test the water vapor permeability and puncture resistance?

MR. FOUQUET: Yes.

RHABDITIDIASIS

J. J. Tancous and W. T. Roddy

Tanners' Council Research Laboratory
University of Cincinnati
Cincinnati, Ohio

Rhabditidiasis, also referred to as Rhabditis dermatitis, is caused by a small worm which burrows in the epidermal portion* making channels through the hide. It is found in "country hides" of animals which have been housed in filthy quarters. The nematode (worm) which causes this disease is normally saprophytic and nonparasitic, living in manure and filth piles where decaying organic matter is present. Under circumstances of extreme insanitation the worm becomes parasitic and burrows into the animal's hide causing a dermatitis and extensive grain damage. In veterinary literature it is reported that if the environment is favorable or, possibly, if animals have nutritional deficiencies, this disease can spread rapidly. The nematodes, as they appear magnified 75 times, are illustrated in Fig. 1.†

An animal having a typical Rhabditis dermatitis is shown in Fig. 2.† The condition involves the area on the leg above the crusted manure. A side of leather made from a hide which was affected by an advanced infestation of Rhabditidiasis is shown in Fig. 3. Tortuous channels are evident; they are characteristic paths taken by the worms when they work their way under the surface of the hide. A magnified view of an area taken from another side of damaged leather and clearly showing the cracks and channels made by the worm is illustrated in Fig. 4.

Cross sections made of leathers damaged by the worms have indicated that they affect only the epidermal area of the hide. Two cross-sectional views of leather showing worms in the tissues are illustrated in Figs. 5 and 6. Figure 5 is a cross-sectional view of a scarified area and shows a worm in the epidermal portion. Figure 6 is a cross-sectional view of a damaged area showing a worm in the structure between the epidermal portion and the corium.

During the past year, 1957, the authors have had several cases of this skin disease brought to their attention for identification. The damage done is permanent, and there is nothing that the tanner can do to correct it. The leather made from hides having advanced infestations is useless.

*The term "epidermal portion" or "epidermal area" is used by the authors to designate that portion of the hide which contains the hair, hair follicles, the epidermis, and its appendages such as the sebaceous glands and the sudoriferous glands, which are surrounded and supported by a collagenous fiber bundle structure. Also, throughout this structure of collagenous fiber bundles are dispersed a network of elastic tissue fibers, erector pili muscles, blood vessels, and nerves.

†Courtesy of E. A. Rhode *et al.* (1). School of Veterinary Medicine, University of California.

FIGURE 1.—The saprophytic nematodes which cause Rhabditis dermatitis. (Courtesy E. A. Rhode *et al.*)

FIGURE 2.—An animal showing areas of Rhabditis dermatitis just above the manure-caked hair. (Courtesy E. A. Rhode *et al.*)

FIGURE 3.—A side of leather made from a hide which was damaged by an advanced infestation of Rhabditidiasis. (Courtesy Fred Rueping Leather Co.)

FIGURE 4.—A magnified view of damaged leather showing cracks and channels made by the worm.

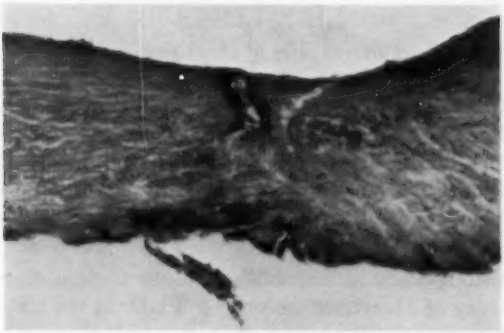

FIGURE 5.—A cross-sectional view of a damaged area showing a worm in the epidermal area.

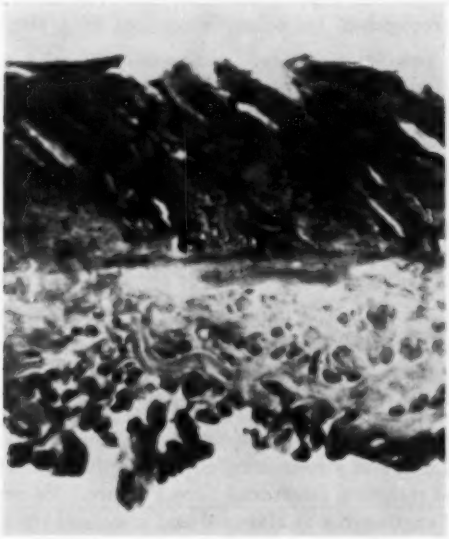

FIGURE 6.—A cross-sectional view of a damaged area showing a worm in the structure between the epidermal area and the corium.

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Received April 5, 1958.

LIFE LINES

Brief Biographies of Our Contributors

BERNARD H. FOUQUET, a new worker in the field of leather chemistry, obtained his Bachelor of Science degree from American University, Washington, D.C., in 1955. The following year he joined the Leather Section of the National Bureau of Standards where he is working on the impregnation of leather with polymers and on other related problems. He joined ALCA in 1957.

DR. JOSEPH R. KANAGY attended Westminster College, Oberlin College, and the University of Maryland, earning his Ph.D. at the last-named institution. On the staff of the National Bureau of Standards for nearly 30 years, he is at present Chief of the Leather Section. Since joining ALCA in 1943 he has been active on several committees and as a member of the Council. He has written numerous papers, many of them published in this journal. The Association recognized his valuable services by giving him the Alsop Award in 1952.

WILLIAM T. RODDY, a graduate of the University of Cincinnati, has been associated with the Tanners' Council Research Laboratory for over 25 years. Since joining ALCA in 1939 he has been an active member, serving on the Physical Testing Committee, the Preparatory and Posttanning Processes Committee, and the Council. His numerous contributions to *JALCA* as well as other journals have added much to our knowledge of the structure of skin, beamhouse processes, tanning, and posttanning operations. Professor Roddy received the coveted Alsop Award at the 1957 ALCA annual meeting.

MRS. JEAN JACOBS TANCOUS holds both B.S. and M.S. degrees from the University of Cincinnati. Since 1949 she has been a Research Associate in the Tanners' Council Research Laboratory at the University, working chiefly on the microscopy and microbiology of skin and hide. She has contributed frequently to this journal.

ALEXIS A. USHAKOFF is a consulting engineer, president of Secotan, Inc., and a member and technical director of Seco Venture. He received his training in mechanical engineering at Harvard and in colloid chemistry at M.I.T. His chief interest has always centered in research and development. He is a recipient of a citation from the Defense Department for scientific contributions to the Air Force and the Navy and is a holder of numerous patents in the areas of machine design, chemical processes, plastic formulation and molding, as well as in the field of solvent tannage and nonaqueous processes for the manufacture of leather and shoes. He has maintained an active interest in this field since 1935. He is a member of ALCA and the American Chemical Society.

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Proceedings, Reports, Notices, and News

of the

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I. U. L. C. S.

The Sixth Biennial Conference of the International Union of Leather Chemists Societies will be held in Munich, Germany, September 6 to 12, 1959.

REPORT OF THE INTERNATIONAL COMMISSION ON THE CHEMICAL ANALYSIS OF LEATHER

The Commission met Sunday September 15, 1957. Those present were: Dr. d'Orsi (Italy), Dr. Gran (Scandinavia), Dr. Grassmann (Germany), Ing. Heyrmans (Netherlands), and Ing. Dumoulin (Belgium), international reporter. Excused were: Ing. Giroflier (France) and Prof. Sagoschen (Austria).

At the meeting in Stockholm the Commission decided to come to a decision about an international method of chemical analysis of leathers, starting with ground leather. The use of a mill for preparing the sample ensures a homogenous sample with a maximum diffusion surface. The study of the different determinations was made systematically, and the following is a résumé of the decisions reached by those present.

Taking the sample.—Adoption of the location chosen by the Committee on Physical Tests.

Preparation of the sample by milling.—Since the degree of disintegration of the leather influences certain determinations, the committee decided to choose an apparatus of the type of the Wiley mill. Other mills based on the same principle—cutting and not grinding of the leather—may be used after having been standardized with respect to the Wiley mill. This control is effected simply by determining water-soluble matter. Three laboratories accepted the task of carrying out this standardization, namely, the laboratory of Highfield Tanning Co., Runcorn, England (Mr. Forsythe); The Federal Swiss Laboratory, St. Gallen, Switzerland (Dr. Engler); and La Stazione Sperimentale per l'Industrie delle Pelli, Naples, Italy (Dr. d'Orsi).

Ash determination.—Three determinations were accepted at Stockholm: soluble ash, insoluble ash, and total ash. All three were made with sulfation to obtain reproducible results. These three determinations being in agreement, it will be enough to make only two determinations—the third result being obtained by addition or subtraction.

Fat determination.—The principal point discussed was the choice of solvent. The Commission recommends the use of chlorinated solvents (methylene chloride or carbon tetrachloride) which assure the extraction of oxidized fatty matter. However, petroleum ether distilling above 60° C. may be used if its use is stated specifically in the analysis report.

Determination of water solubles.—This determination being necessarily arbitrary, the Commission considers it logical to choose the simplest method from among those that give reproducible results on ground leather. This is the method of cold agitation, similar to the former official method that was

applied to leather cut into strips. The results obtained are of the same order of magnitude. The principal advantage is the reduction of the time of agitation from 5 to 2 hr. at a temperature of 25° C. As a result the Commission proposes the provisional adoption of this method. The opinions of the various societies should be sent to Dr. Engler, international reporter for the next term, before May 31, 1958.

Determination of hide substance.—The introduction of extraneous nitrogenous matter into leather has completely falsified the usual method. The Commission earnestly asks that you send in proposals of methods that will give significance to this determination as well as to the indices that are derived from it.

pH determination.—An international method will be proposed at the next meeting after examination of the methods now used in different member countries of the Union.

The Commission requests the publication of this report and of the officially adopted methods in the journals of the various national associations.

In terminating my duties as international reporter of the Commission for Chemical Analysis of Leathers, I thank all the national reporters who greatly helped me in my task, and I hope that Dr. Engler, our new international reporter, will have the benefit of as complete a collaboration. After January 1, 1958, the international reporter of the International Commission on the Chemical Analysis of Leather will be Dr. Engler, Understrasse 11, St. Gall, Switzerland.

Rome, September 20, 1957 PROF. ING. V. DUMOULIN

WORKING METHODS ADOPTED AT THE ROME MEETING

I. **Preparation of the Sample for Analysis.**—As published in *J. Soc. Leather Trades' Chemists*, **40**, 59 (1956).

II. **Determination of Water.**—Dry 5 g. of ground leather to constant weight, in a receptacle about 50 mm. in diameter, in an ordinary oven or a vacuum oven, at a temperature of 102° ± 2° C. Make the first weighing after 3 hr. of drying. Consider the weight to be constant when two consecutive weights, at a one-hour interval, do not differ by more than 5 mg.

III. **Determination of Ash.**—(*a*) *Total sulfated ash*: As published in *J. Soc. Leather Trades' Chemists*, **40**, 62 (1956). (*b*) *Sulfated soluble ash*: As published *Ibid.*, **40**, 63 (1956). (*c*) *Sulfated insoluble ash*: After the extraction of matter soluble in water, dry the leather in the air, then transfer it carefully to a tared crucible. Heat slowly at the beginning until the leather decomposes. Continue in the muffle furnace and sulfate as for total ash.

IV. **Determination of Matter Soluble in water.**—As published in *J. Soc. Leather Trades' Chemists*, **40**, 89 (1956).

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AUDIT REPORT

February 18, 1958

THE AMERICAN LEATHER CHEMISTS ASSOCIATION, INC.
THE UNIVERSITY OF CINCINNATI
CINCINNATI, OHIO

GENTLEMEN:

In accordance with the request of Dr. Fred O'Flaherty, Secretary-Treasurer, we have examined the balance sheet of the American Leather Chemists Association, Incorporation, as of December 31, 1957, and the related statements of income and surplus for the year then ended. Our examination was made in accordance with generally accepted auditing standards, and accordingly included such tests of the accounting records and such other auditing procedures as we considered necessary in the circumstances.

The report of the examination is presented herewith, together with the following exhibits:

EXHIBIT I: Balance Sheet as of December 31, 1957, and comparison with December 31, 1956.

EXHIBIT II: Statement of Income for the Year ended December 31, 1957, and comparison with the year ended December 31, 1956.

EXHIBIT III: Statement of Surplus for the Year ended December 31, 1957, and comparison with the year ended December 31, 1956.

The commercial account balance on deposit with the Central Trust Company was confirmed independently and was reconciled with the cash account balance of the Association.

Accounts receivable were not circularized; this procedure was considered unnecessary in view of the small balance of each account, and in view of the fact that of the total accounts receivable of $1,827.01 at December 31, 1957, approximately eighty-five per cent of the total was collected in January, 1958. By other auditing procedures, we have satisfied ourselves of the accuracy, validity, and collectibility of the accounts.

The investment securities were confirmed by Dr. Fred O'Flaherty. In addition, we examined the bonds in detail. All bonds are in the safe deposit box at the Central Trust Company.

There were no bond purchases or sales in 1957. Accumulated interest on the Series F bonds has been added to the original cost of the $5,000.00 par value of Series F bonds owned at December 31, 1957.

At December 31, 1957, the total accumulated value of the Series F bonds is $4,500.00, of which $800.00 represents accumulated interest. In the year 1957, all bond investments earned interest of $1,205.00.

Dues received in advance totaling $195.50 and subscriptions received in advance totaling $3,114.42 were verified in detail and were reconciled with the controlling accounts.

Net income for the year ended December 31, 1957, was $1,168.98, compared with $1,506.76 for the year ended December 31, 1956, or a decrease of $337.78. The causes of the decreased net income are analyzed in Exhibit II. In the year 1957, revenues increased $1,309.93, and expenses increased $1,647.71, due primarily to increased printing costs. The net income is satisfactory.

In our opinion the accompanying balance sheet and related statements of income and surplus present fairly the financial position of the American Leather Chemists Association, Incorporated, at December 31, 1957, and the results of its operations for the year then ended, in conformity with generally accepted principles of accounting, applied on a basis consistent with that of the preceding year.

Respectfully submitted,

ARTHUR W. HOLMES
Certified Public Accountant

EXHIBIT I

BALANCE SHEET AT DECEMBER 31, 1957 AND COMPARISON WITH DECEMBER 31, 1956

	December 31, 1957	December 31, 1956	Increase or Decrease* 1957 over 1956
ASSETS			
Current Assets:			
Cash on hand	$ 1.60	$ 24.16	$ 22.56*
Cash in bank	16,289.76	16,007.48	282.28
Accrued interest on Treasury Bonds	537.50	62.50	475.00
Accounts receivable, Directory	–0–	47.00	47.00*
Accounts receivable, advertisers	1,714.51	1,846.35	131.84*
Accounts receivable, reprints	112.50	–0–	112.50
Total Current Assets	$18,655.87	$17,987.49	$ 668.38
Investments:			
U. S. Savings Bonds, series F, (cost $3,700.00 plus $900.00 accrued interest at December 31, 1957)	$ 4,500.00	$ 4,370.00	$ 130.00
U. S. Treasury Bonds, 2.5%, 1972–1967, at cost	28,495.00	28,495.00	–0–
U. S. Treasury Bonds, 2.5%, series G, due July, 1958; at cost	5,000.00	5,000.00	–0–
U. S. Treasury Bonds, 2.5%, 1968–1963, at cost	8,842.50	8,842.50	–0–
Total Investments	$46,837.50	$46,707.50	$ 130.00
Total Assets	$65,493.37	$64,694.99	$ 798.38
LIABILITIES AND CAPITAL			
Current Liabilities:			
Accounts payable	$ 1,917.01	$ 80.46	$1,836.55
Income tax withheld	237.40	194.80	42.60
City tax withheld	14.99	13.00	1.99
Total Current Liabilities	$ 2,169.40	$ 288.26	$1,881.14
Deferred Credits to Income:			
Advertising received in advance	$ 24.00	$ –0–	$ 24.00
Dues received in advance	195.50	388.00	192.50*
Subscriptions received in advance	3,114.42	3,197.66	83.24*
Total Deferred Credits to Income	$ 3,333.92	$ 3,585.66	$ 251.74*
Total Liabilities	$ 5,503.32	$ 3,873.92	$1,629.40
Capital:			
Earned surplus (Exhibit III)	$58,990.05	$58,821.07	$ 168.98
Reserve for foreign meetings	1,000.00	2,000.00	1,000.00*
Total Capital	$59,990.05	$60,821.07	$ 831.02*
Total Capital and Liabilities	$65,493.37	$64,694.99	$ 798.38

EXHIBIT II

STATEMENT OF INCOME

For the years ended December 31, 1957 and December 31, 1956

	December 31, 1957	December 31, 1956	Increase or Decrease* 1957 over 1956
Revenues, General:			
Dues, *Journal* subscription included	$11,592.00	$11,489.01	$ 102.99
By-Laws and Methods booklets	73.00	82.57	9.57*
Directory	770.66	804.20	33.54*
Interest income	1,205.00	1,205.00	–0–
Total General Revenues	$13,640.66	$13,580.78	$ 59.88
Revenues, *Journal*			
Advertising	$12,326.68	$11,348.19	$ 978.49
Subscriptions	6,223.39	5,448.88	774.51
Journal sales	555.52	1,107.70	552.18*
Reprints	1,299.97	1,388.49	88.52*
Dues, I.S.L.T.C.	943.75	806.00	137.75
Total *Journal* Revenues	$21,349.31	$20,099.26	$1,250.05
Total Revenues	$34,989.97	$33,680.04	$1,309.93
Expenses, General:			
Executive secretary expense	$ 4,600.00	$ 4,200.00	$ 400.00
Office operating expense	1,087.34	1,350.90	263.56*
Uncollectible accounts expense	323.00	476.00	153.00*
Directory expense	894.67	912.19	17.52*
Annual meeting expense	275.16	218.07	57.09
Council meeting expense	158.90	84.35	74.55
Auditing	200.00	200.00	–0–
Winheim Memorial Award	100.00	–0–	100.00
Leather Chemists Union Dues	119.25	119.28	.03*
By-Laws and Methods	–0–	361.35	361.35*
Total General Expenses	$ 7,758.32	$ 7,922.14	$ 163.82*
Expenses, *Journal*:			
Printing	$19,921.15	$18,082.43	$1,838.72
Managing editor, compensation	2,500.00	2,083.33	416.67
Managing editor, expense	191.05	120.35	70.70
Editor-in-chief, compensation	600.00	500.00	100.00
Editor-in-chief, expense	199.64	358.38	158.74*
Editor's compensation	–0–	350.00	350.00*
Editor's expense	–0–	57.70	57.70*
Storage	350.00	350.00	–0–
Insurance	62.00	62.00	–0–
Miscellaneous	–0–	60.00	60.00*

Copyright registration	48.00	48.00	–0–
Reprints	1,667.43	1,640.00	27.43
Exchange charges	5.35	1.05	4.30
Translations and abstracts	294.19	305.08	10.89*
Committee expense	97.80	109.25	11.45*
Advertising discounts allowed	126.06	123.57	2.49
Total *Journal* Expense	$26,062.67	$24,251.14	$1,811.53
Total Expenses	$33,820.99	$32,173.28	$1,647.71
Net Income	$ 1,168.98	$ 1,506.76	$ 337.78*
Net Income distributed:			
General	$ 5,882.34	$ 5,658.64	$ 223.70
Journal	(4,713.36)	(4,151.88)	561.48
Total	$ 1,168.98	$ 1,506.76	$ 337.78*

EXHIBIT III

STATEMENT OF SURPLUS

For the years ended December 31, 1957 and December 31, 1956

	Year Ended December 31, 1957	Year Ended December 31, 1956	Increase or Decrease* 1957 over 1956
Balance, January 1	$58,821.07	$59,314.31	$ 493.24*
Add Net Income:			
General	5,882.34	5,658.64	223.70
	$64,703.41	$64,972.95	$ 269.54*
Deduct Net Loss:			
Journal	$4,713.36	$ 4,151.88	$ 561.48
	$59,990.05	$60,821.07	$ 831.02*
Deduct:			
Allocation to reserve for foreign meetings	1,000.00	2,000.00	1,000.00*
Balance, December 31	$58,990.05	$58,821.07	$ 168.98

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E. Lovendahl Jensen, Teknologisk Institut, Garverforeningens Forsogsstation, Hagemannsgade 2, Copenhagen V, Denmark.

Edward F. Kelly, 116 Sutherland Road, Apt. No. 2, Brighton, Massachusetts.

Earl W. Loucks, Thiokol Chemical Corporation, 780 N. Clinton Avenue, Trenton, New Jersey.

Edwin L. Quinn, K. J. Quinn & Company, 195 Canal Street, Malden 48, Massachusetts.

John W. Quinn, Jr., K. J. Quinn & Company, Inc. (Del.), 606 West Oregon Street, Milwaukee 4, Wisconsin.

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E. H. Cox, Box 37, Camden, Maine.

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Edward J. Delaney, 78 Percy Street, Dracut, Massachusetts.

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Stoddard Martin, 523 Slayton, Grand Haven, Michigan.

Lauren E. Meyers, 6060 N. Berkeley Blvd., Milwaukee 17, Wisconsin.
T. Joseph O'Connor, 17 Stafford Road, Danvers, Massachusetts.
Joseph A. Paciello, 328 Sugartown Road, Devon, Pennsylvania.
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Gene W. Stanbery, 640 Bays View Road, Kingsport, Tennessee.
James H. Tirrell, 26 Florence Avenue, Lowell, Massachusetts.
John D. Tomlinson, 2250 Brimley Road, Agincourt, Ontario, Canada.
Herman P. Wagner, c/o Poetsch and Peterson, 325 South Maple Avenue, South San Francisco, California.
Jack Zeitlin, 1873 48th Street, Brooklyn 4, New York.

ABSTRACTS

Making Use of the Tannin in Oak Galls. G. Vago. *Das Leder*, **8**, 199–200 (1957).—Hungarian galls which occur on leaves of the oak, *Quercus pedunculata*, have found only limited use. Fresh galls contain 9–13% tannin with a purity of 80–85 by the filter method or 70% by the shake method. Tests indicate that the tannin is a mixture of the condensed and hydrolyzable types. The liquors have an unusually high viscosity; sludge forms if they are concentrated to 3–4% Bé., and they gel completely at a concentration of 8–10%. Investigations were made of the cause of sludge formation. The galls contained 1.8% ash, but the content of heavy metals was low and could not cause sludge. Fermenting the sugar and removing pectin with enzymes did not prevent the formation of sludge. Roasting the galls for 4 hr. at 120°–125°C. before extraction, to denature proteins, increased the amount of sludge. Purification of the tannin by precipitating it with lime and then redissolving it in sulfuric acid, sulfiting the extract, and use of nonionic or anionic emulsifiers were not effective in preventing gelation. No differences in sludging were found between fractions when galls were leached at 20°C. and several different fractions were collected and tested. The above results led to the conclusion that the extractive consisted of relatively high molecular compounds, of high astringency, which aggregated to gels during concentration of the solution. By evaporation of 2° Bé. liquors in a vacuum after they had been adjusted to various pH values, clear solutions of 8°–10° Bé. were obtained from the liquors adjusted to pH 5.5–7, and the solutions did not gel for several days. Solutions from liquors adjusted to pH 7.1–7.5 were stable for months. Dark-colored solutions were obtained at pH values above 8. A 23°-Bé. extract made from a liquor adjusted to pH 7.2 was stable for 6 months and clear. Even after evaporation to dryness it would redissolve to a clear solution that could be acidified without sludge formation to pH 4.7 with acetic acid but not with mineral acid. A similar

extract was made by leaching galls with 0.3% $NaHCO_3$ solution. The best leaching temperature for the galls was 70°C. The extract could be used in vegetable tanning blends and for retanning chrome leather. In spite of its high pH value the extract had excellent astringency and made leather that was light-stable and not too dark. I.D.C.

PATENTS

Method for Treating Leather. U. S. Pat. 2,824,816. Ian C. Somerville, Willow Grove, and Richard F. M. Fisher, Jenkintown, Pa., assignors to Rohm & Haas Company, Philadelphia, Pa., a corporation of Delaware. Appl. May 13, 1954.—1. The method of treating leather comprising agitating the air-dry leather with a solution in a volatile hydrophobic organic solvent medium of a hydrophobic impregnant until substantial impregnation is effected, adding a water-soluble hydrophilic solvent selected from the group consisting of volatile organic solvent and aqueous solutions of volatile organic solvents comprising at least a predominant proportion of organic solvent in which the impregnant has substantially less solubility as compared to the hydrophobic solvent while continuing the agitation until additional impregnation is effected, removing the leather from the liquid system, draining it, and drying it.

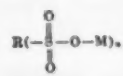
Method of Producing a New Material from Waste Leather. U. S. Pat. 2,827,413. Yehuda Friedmann, Tel Aviv, Israel. Appl. Sept. 20, 1955. Claims priority, appl. Israel, Sept. 21, 1954.—1. The method of producing a new sheet material from small waste leather pieces, comprising the steps of cutting discs of uniform size from said waste leather pieces, superposing said discs

onto one other and into stack formation, bonding said discs to each other while so superposed to thereby form a rod of leather, spirally cutting said rod longitudinally of the latter to form a multistrip sheet of leather, superposing a plurality of similarly formed sheets on one another with the direction of said strips in each sheet running transversely to the direction of the strips in each adjacent sheet, and bonding said sheets to one another while so superposed to thereby form a composite multiple-sheet material of enhanced strength and workability characteristics.

Process of Production of Tanning Agents in Solid Form. U. S. Pat. 2,801,986. Martin Meister, Leverkusen-Wiesdorf, and Gustav Mauthe, Opladen,

Germany, assignors to Farbenfabriken Bayer Aktiengesellschaft, Leverkusen, Germany, a corporation of Germany. Appl. Jan. 15, 1953. Claims priority, appl. Germany, Jan. 18, 1952.—12. The process of preparing tanning agents in solid form which comprises reacting in a concentrated aqueous solution of a salt of the group consisting of alkali halides and alkali nitrates at a temperature up to about 100°C., a mixture containing a polyhydric phenol, a hydrochloride of an aromatic amine, and an aliphatic saturated aldehyde at least until the precipitate is water-soluble.

Imparting Water Resistance to Leather. U. S. Pat. 2,827,432. Alfred W. Hopton, Lewiston, N. Y. No drawing. Appl. July 8, 1954.—1. Process of rendering leather water-resistant, comprising impregnating said leather with a leather-stuffing composition including at least one member of the group consisting of fat, oil, wax and petrolatum, and containing as an activating ingredient, in an amount from about 4 percent to about 75 percent by weight of said composition, a high-molecular-weight organic compound characterized by a hydrocarbon radical having at least about 27 carbon atoms, said high-molecular-weight organic compound being a member of the group consisting of (1) sterols having at least about 27 carbon atoms in the molecule, and (2) a water-insoluble sulfonate of a molecular weight of at least about 470 and having the general formula



wherein R is a hydrocarbon radical having at least about 27 carbon atoms, M is a member of the group consisting of monovalent metal and amino, and n is at least 1, the ratio of the numerical value of n to the number of carbon atoms in R being not more than about 1:27.

Process of Tanning Skins with Acidic Solutions of Alkali Metal Zirconium Silicates. U. S. Pat. 2,826,477. William J. Rau, Glenside, and Ian C. Somerville, Willow Grove, Pa., assignors to Rohm & Haas Company, Philadelphia, Pa., a corporation of Delaware. Appl. May 18, 1954.—1. A process of tanning a skin comprising tanning the skin in an aqueous solution of an acidic material selected from the group consisting of sulfuric acid, hydrochloric acid, nitric acid, sodium acid sulfate, and sulfamic acid, at a temperature not over 35°C., containing suspended therein a pulverized water-insoluble double silicate of zirconium and an alkali metal M in which the molar ratio SiO_2:ZrO_2 is from about 1:1 up to but not over 1.5:1 and the molar ratio SiO_2:M_2O is from about 1:1 up to but not over 3:1, the acidity being adjusted to provide the corresponding salt of zirconium of zero to 60% basicity calculated on the Schorlemmer scale upon complete dissociation of the sodium zirconium silicate, the skin remaining in this tanning liquor until it is tanned to the desired extent.

Tanning Resin. Brit. Pat. 787,347. Farbenfabriken Bayer Akt.-Ges. Appl. Jan. 26, 1955.—Solid water-soluble kneadable tanning resins are prepared by mixing sulphite waste liquor which has been treated with caustic alkali, with condensation products of aromatic sulphonic acids with aromatic hydroxy compounds and aldehydes, or condensation products of aromatic compounds containing sulphomethyl groups with aromatic hydroxy compounds and aldehydes, or incompletely sulphonated or sulphomethylated condensation products of aromatic hydroxy compounds with aldehydes, and thereafter precipitating the water-soluble resin by means of a mineral acid or an alkali metal or ammonium salt thereof. According to examples: (1) beta-naphthol was sulphonated with sulphuric acid monohydrate at 115°C. and the product stirred with phenol, cresol and water. The mixture was subsequently condensed with formaldehyde and to the condensation mixture there was added sulphite waste liquor which had previously been boiled with caustic soda, together with a minor amount of a moist resin obtained by boiling cresol with formaldehyde in dilute sulphuric acid and drawing off the supernatant acid. The mixture of condensation product, alkali-pretreated sulphite waste liquor and cresol-formaldehyde resin was adjusted to *p*H 3.1 with sulphuric acid and the tanning resin recovered by the addition of ammonium sulphate and common salt. (2) Dihydroxydiphenylsulphone was homogenized with naphthalene which had been sulphonated with sulphuric acid and the mixture condensed with 30 per cent formaldehyde at 105°C. To the condensation mixture were added sulphite waste liquor which had been treated with caustic soda, and a minor amount of a moist phenol-formaldehyde resin. The *p*H was adjusted to 3.5 with aqueous ammonia and the tanning resin salted-out with ammonium sulphate and common salt. (3) Naphthalene was sulphonated with sulphuric acid monohydrate and the sulphonated mixture was condensed with formaldehyde at 105°C.; the condensation mixture was thereafter diluted with water and after the addition of crude pyrocatechol, was further condensed with formaldehyde. Caustic soda-treated sulphite waste liquor was then added and the tanning resin precipitated with ammonium sulphate and common salt. (4) Naphthalene was sulphonated with sulphuric acid and mixed with ortho-chlorophenol and formaldehyde. Sulphite waste liquor, pretreated with caustic soda, was added to the mixed condensation product and the mixture rendered acid by concentrated hydrochloric acid; the tanning resin was precipitated with ammonium sulphate. (5) A mixture of naphthalene and a minor portion of beta naphthol was sulphonated at below 120°C. with sulphuric acid and the mixture after diluting with water was condensed with formaldehyde at 100°C. Separately from this mixture, a mixture of phenol and cresol was condensed with formaldehyde in the presence of sodium sulphite at 100°C. The two condensation products were thereafter mixed at 75°C. and the mixture stirred with caustic soda pre-treated sulphite waste liquor, and the tanning resin precipitated by the addition of a saturated solution of ammonium sulphate and common salt. (6) Into the mixture of the condensation product of naphthalene sulphonic acid and the condensation product of phenol-cresol and sodium sulphite of Example (5) above, there was introduced the reaction product of resorcinol and an aldol-acetaldehyde mixture and formic acid. Caustic soda pretreated sulphite waste liquor was added to the mixture and the product finally salted-out with saturated ammonium sulphate solution and common salt. The invention is stated to be applicable to other aryl sulphonic acids, for example substituted naphthalene sulphonic acids, benzene sulphonic acid, anthracene

sulphonic acids, phenanthracene sulphonic acids or substitution products thereof as well as sulphomethyl compounds of the same type. As aromatic hydroxy compounds there may be used mono-, di- or tri-hydroxy benzenes or naphthalenes which may be further substituted by alkyl, halogen or sulpho groups. Sulphonated compounds of the Novolac type may be employed as partially sulphonated condensation products. Specifications 467,998, [*Group* VI], and 496,898 are referred to.

Zirconium Tannage. Brit. Pat. 786,769. Rohm & Haas Co. Appl. April 29, 1955.—Skins are tanned at not over 35°C. in an aqueous acid suspension of a pulverized zirconium-alkali metal silicate, said silicate having a molar ratio SiO_2:ZrO_2 not over 1.5:1 and SiO_2:M_2O not over 3:1, where M is the alkali metal. The process may be employed alone, before or after chrome, iron, aluminium, vegetable, synthetic or formaldehyde tanning, or in a single stage combination tannage with a mineral tanning agent such as chromium. The tanning liquor is conveniently prepared from a dry pulverized composition comprising a solid acid such as sodium bisulphate or sulphamic acid and the desired silicate wherein the amount of acid is such as to provide after complete reaction with the silicate a salt of zirconium of 0—60 per cent basicity. Preferred double silicates have the formulae Na_2ZrSiO_5 and $Na_4Zr_2Si_3O_{12}$ (*see* Group III); other alkali metals referred to are potassium and lithium. Other specified acids are sulphuric, nitric, hydrochloric and nitric cake. A salt such as sodium chloride or sulphate should be present, e.g. to an extent of 3—10 per cent in the tanning liquor before the skin is introduced; thereafter the skin may be added followed by the silicate and acid components in either order or the skin may be added, preferably promptly, after the incorporation of the ingredients in the liquor. Based on the weight of the skin, 12—15 per cent silicate gives a full tan: it may be added batchwise if desired. The process may be applied to any skins, but preferably pickled skins having a *p*H of 1.5—2 are used. In the examples, pickled calfskins were added to liquors and a *p*H of 1.5—2 obtained finally. They were then neutralized to *p*H 4.5—4.75 with sodium acetate and bicarbonate and washed, fat-liquored and finished conventionally.

Washing Furs. Brit. Pat. 787,138. Boehme Fettchemie Ges. Appl. Aug. 12, 1955.—Chrome-tanned pelts are contacted with an aqueous liquor containing a water-soluble complex-forming compound having a carboxyl, hydroxyl or phosphoric acid group and a lipophilic residue to remove chromium soaps from the wool or hair of said pelt. The lipophilic group preferably has 10–18 carbon atoms and a group conferring water solubility such as an acid sulphuric acid ester, a sulphonic acid or a polyalkylene oxide group, and the liquor may additionally contain a surface-active compound such as a conventional pelt washing agent, e.g. alkyl sulphates, alkyl sulphonates or alkyl benzene sulphonates. The pelt also may be washed subsequently with a conventional neutral pelt washing agent. Compounds referred to are water-soluble mono-esters of di- and poly-basic inorganic or organic acids (phosphoric, tartaric, citric) with higher molecular weight hyroxyl compounds, cycloaliphatic (naphthene) or aliphatic-aromatic (alkylphenols) alcohols and their alkoxylation products, or their sodium or ammonium salts; and the polycarboxylic acids formed by

reaction of unsaturated fatty acids (oleic, linseed oil, soya oil fatty acids) with maleic acid; their salts and derivatives (by sulphonation or ethoxylation) may also be used if they contain at least one carboxyl group capable of complex formation. In the example, chrome tanned sheepskin fleeces were treated at 40–50° C. with a mixture of the sodium salts of monophosphoric acid esters of ethoxylated C_{12}–C_{18} alcohols and a sodium alkyl sulphate mixture. They were subsequently washed in an aqueous ammonium alkyl sulphate mixture.

Making Leather. Brit. Pat. 768,762. Farbwerke Hoechst Akt.-Ges. Vorm. Meister Lucius & Bruning. Appl. April 7, 1955.—Tanning or retanning is effected using an agent made by homogeneously mixing a melted salted-out condensation product of a dihydroxy diaryl sulphone, an alkyl or aralkyl sulphonic acid and formaldehyde with an aluminium salt rendered partially basic and partially masked with an organic acid and water is evaporated from the mixture, said aluminium salt being added in such an amount and the water evaporated to such an extent that the melt solidifies on cooling (*see* Group IV (a)). In Examples (1) and (2), a condensation product from naphthalene sulphonic acids, 4 : 4¹-dihydroxy-diphenyl sulphone and formaldehyde containing free sulphuric acid is salted out with sodium chloride solution, the sodium salt of the tanning agent being separated and mixed with (1) sodium oxalate, sodium acetate and aluminium sulphate; (2) oxalic acid, citric acid, aluminium sulphate, glacial acetic acid, formic acid and sodium hydroxide. In Examples (3), (4) and (5):—(3) bated and bleached calf pelt is suspended in an aqueous solution of the tanning agent of Example 1 for 8 days and fat liquored with a sulphonated oil to give nubuk or white upper leather; (4) chrome-tanned neutralized box leather is retanned with a solution of the tanning agent of Example 2 for 2 hours and fat liquored with a sulphamido-acetic acid of a synthetic paraffin of medium chain length; (5) python skins pretanned with formaldehyde are further tanned for 5 days with a solution of the tanning agent of Example 2 and fat liquored.

Tanning Compositions. Brit. Pat. 785,474. Boehme Fettchemie Ges. Appl. March 21, 1955.—An oiling and tanning liquor for animal hides and skins is made by mixing sulphonated sperm oil, sodium alkyl sulphate and a condensation product obtained by heating a polyethylene oxide of average molecular weight 9000 with a reaction product of linoleic acid and maleic anhydride (*see* Group IV (a)), heating the mixture and forming the product into an aqueous emulsion to which are added chrome tanning salts and common salt.

Coated Leather. Brit Pat. 769,730. Farbenfabriken Bayer Akt.-Ges. Appl. Jan. 20, 1955.—Leather is dressed with an aqueous emulsion comprising a copolymer of butadiene and acrylonitrile, the molecular weight of said copolymer having being kept so low by the addition of a chain terminating substance that when isolated the copolymer has an oily-viscous consistency such that its hardness cannot be measured with a Defo measuring instrument. The product is stated to be not tacky. Optionally a third vinyl component, e.g. styrene, substituted styrenes, acrylic ester, methacrylic ester and asym. dichloroethene may be included. If desired, the emulsion may be mixed with emulsions of polymers

having a Defo hardness of e.g. 6,000. The copolymer preferably should not contain more than 50 per cent by weight of acrylonitrile. The copolymer may be applied, e.g. by the collodion dressing method or as exemplified by brushing (or spraying) with a coating dye. In the example a buffed semi-chrome tanned cowhide is brushed to remove dust and brushed with an aqueous liquor comprising a casein-based coating dye, a viscous-oily butadiene/acrylonitrile copolymer and an aniline dyestuff. After absorbing this "bottoming," the leather is brushed with an aqueous liquor comprising a casein-based coating dye, an aniline dyestuff, a specified butadiene/acrylonitrile copolymer, an oily viscous butadiene/acrylonitrile copolymer and an ammonia-saponified casein solution. The coated leather is then dried and ironed on a hydraulic press at 60° C. and 200 atmospheres. The product is then sprayed with a further quantity of the liquor after dilution and coated with a collodion lacquer which is hardened and the leather finally dried and ironed at 60°C. and 150 atmospheres. In place of the collodion lacquer, an albumen-casein wax-based ironing glaze could have been used. The butadiene-acrylonitrile copolymer used is made by dissolving sodium salts of sulphonic acids of long-chain paraffins, sodium hydroxide, sodium pyrophosphate and potassium persulphate in water, and emulsifying therein butadiene (60 parts) and acrylonitrile (40 parts), the latter containing 6 parts of diisopropylxanthic disulphide as the chain terminating agent; triethanolamine is added and the mixture polymerized at 40° C. until about 80 per cent of the monomers had reacted; di-tert-butyl-1.5-*p*-cresol is then added as a stabilizer. An alternative copolymer specified contains styrene in addition to butadiene and acrylonitrile, and dodecyl mercaptan as the chain terminating agent. Specifications 633,631 and 678,614 are referred to.

BOOK REVIEW

Wood Tanks Reference and Data Handbook. National Wood Tank Institute, 332 S. Michigan Ave., Chicago, Ill. 27 pp. No charge.—This pamphlet contains many useful data on the design, construction, care, and use of wood tanks; properties of woods; and possible tank linings. H.B.M.

CORRECTIONS

In the February, 1958, number of this journal, please note the following corrections pertaining to the paper by Seligsberger and Clayton entitled "The Action of Acrylonitrile and Acrylamide upon Collagen":

p. 98, 5th line from bottom : change (14) to (13).

p. 102, reference 13 : add "see also *The Chemistry of Tanning Processes* (New York : Academic Press, 1956) p. 88."

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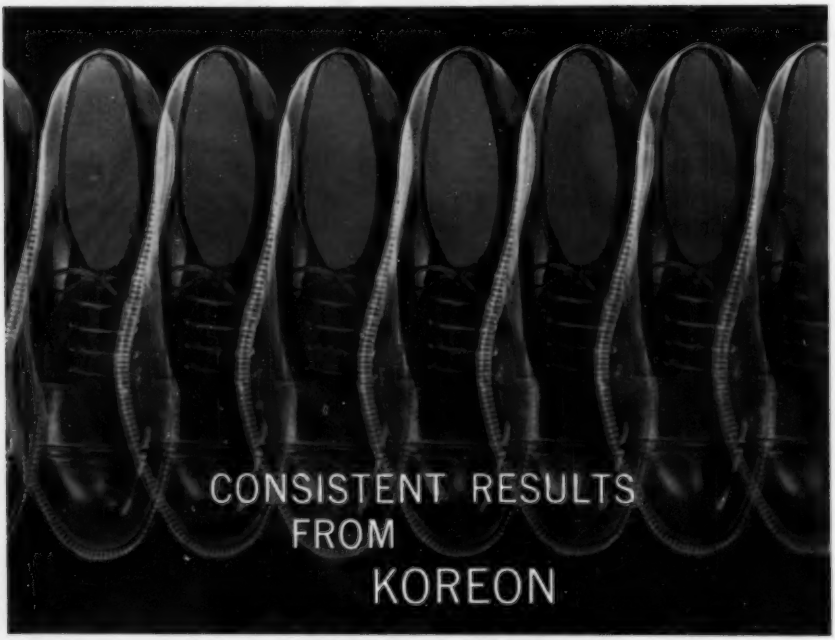

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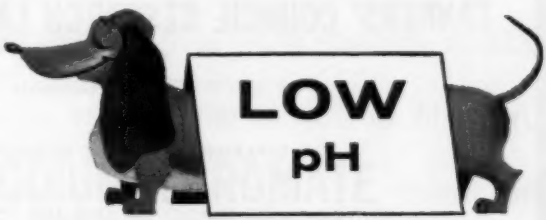

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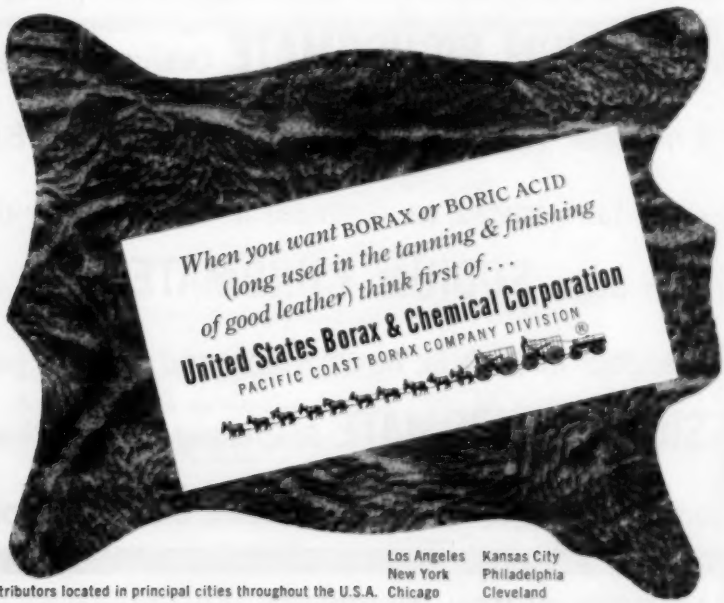
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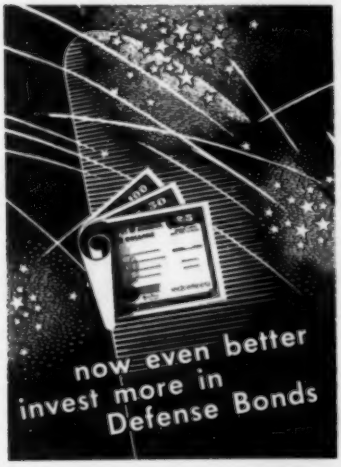

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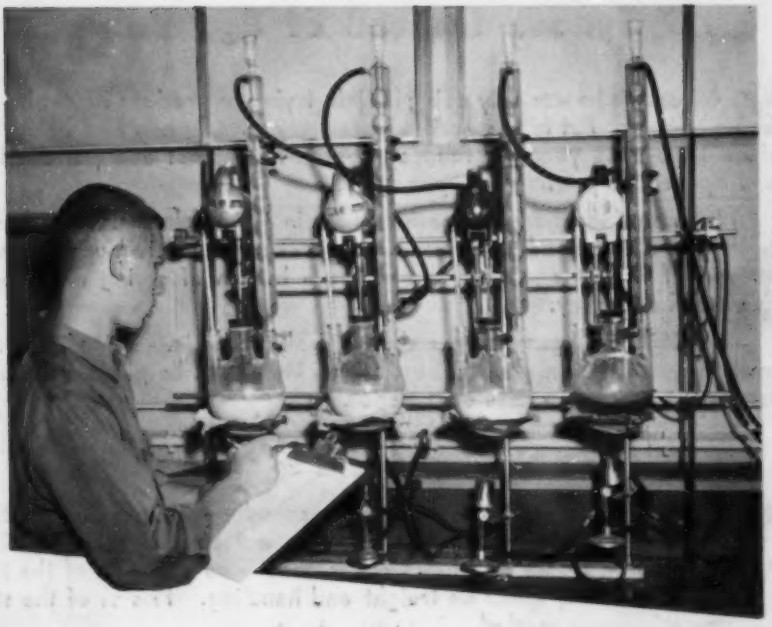

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